Tappi

PUBLISHED BY THE TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY

NOVEMBER 1952

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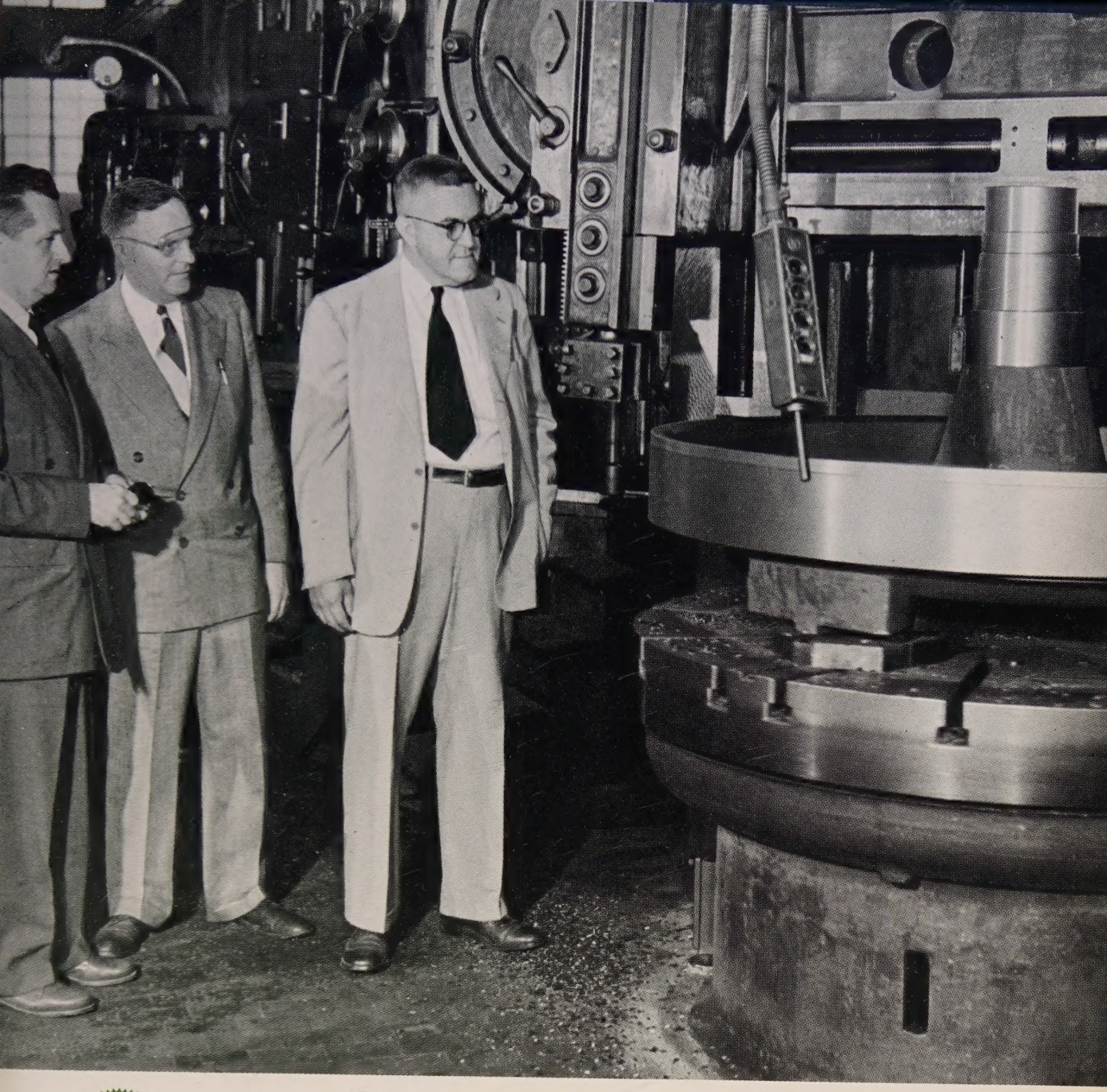

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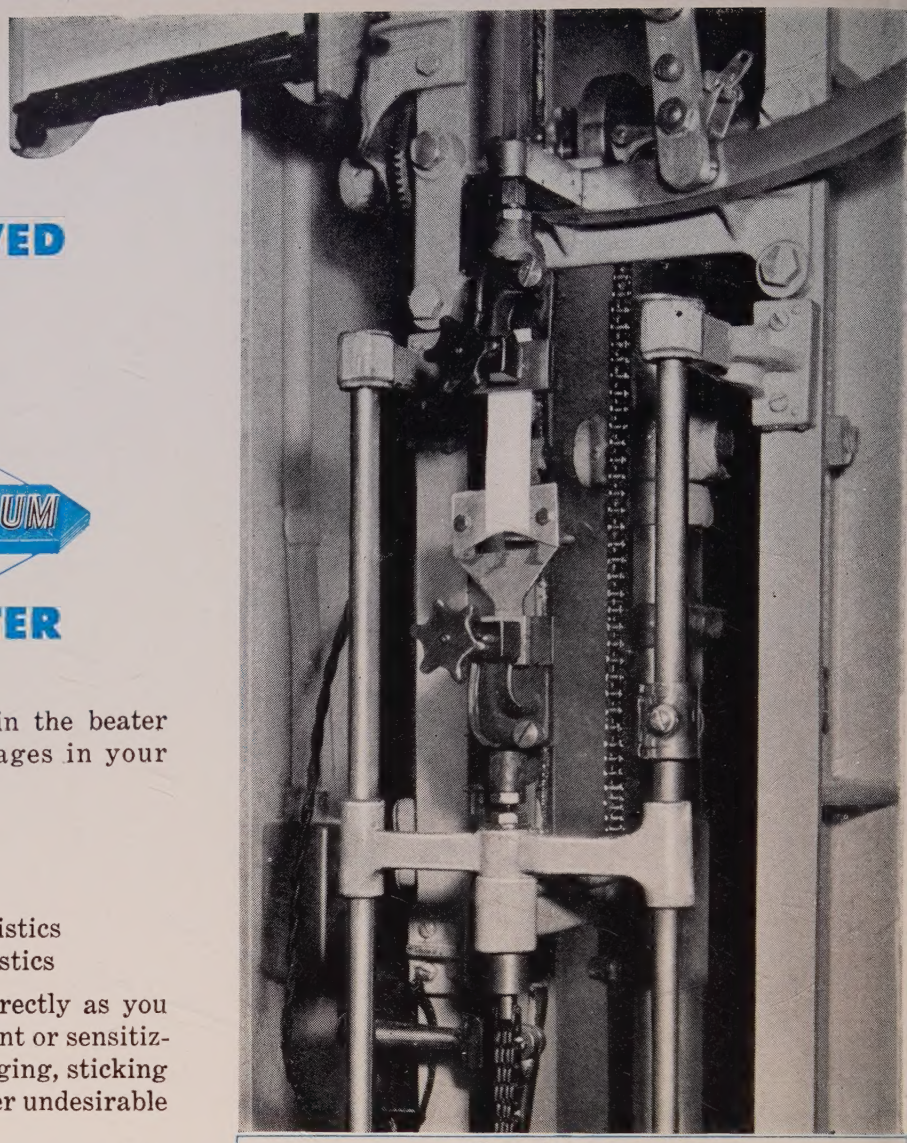

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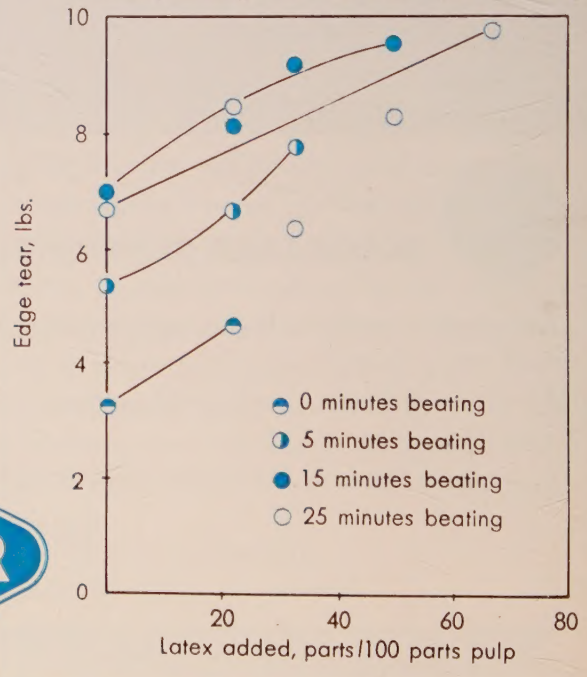

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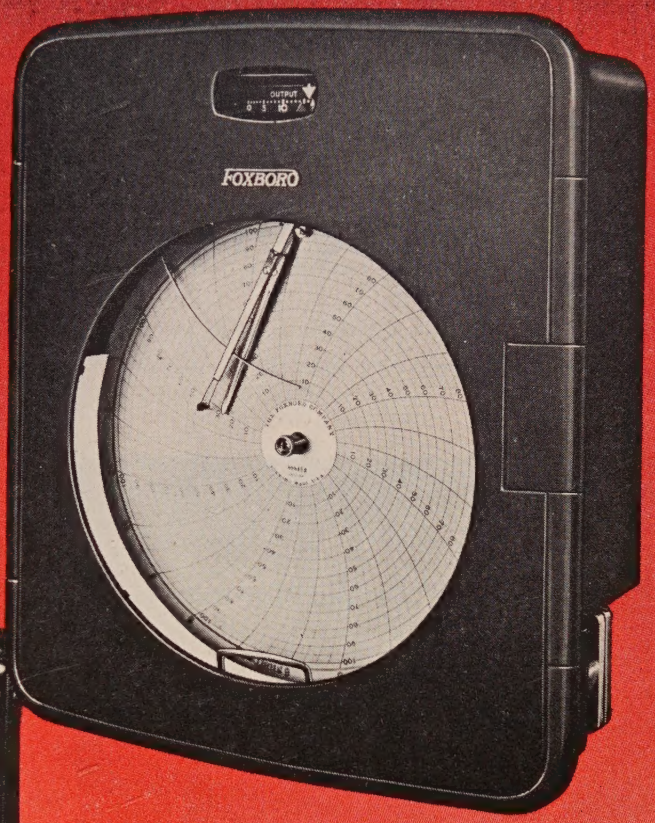

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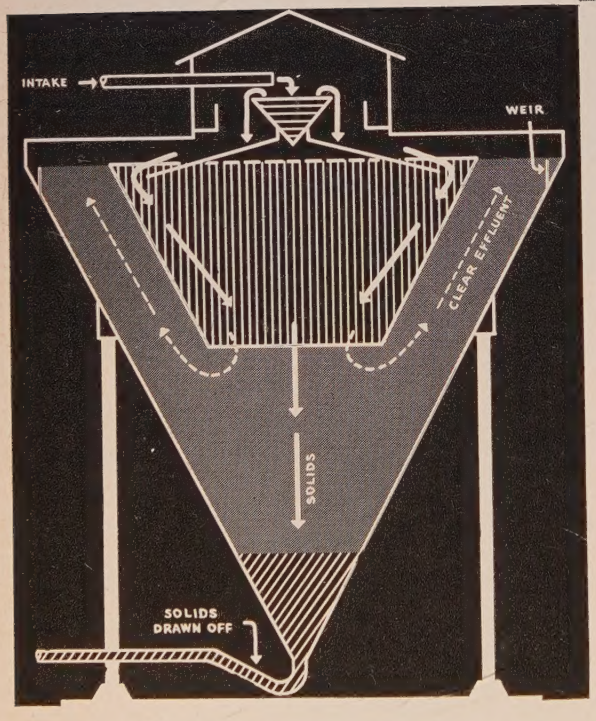

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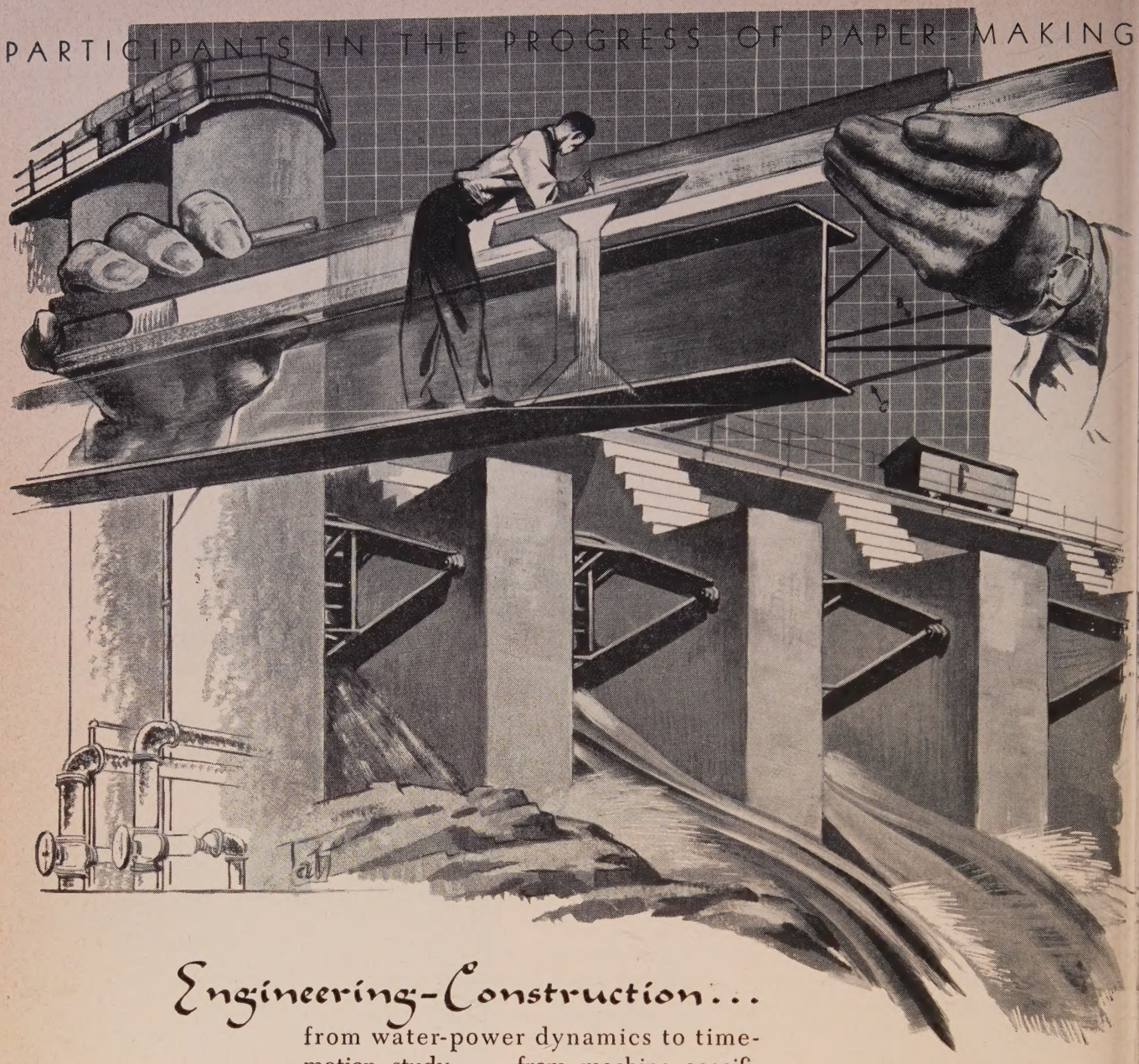

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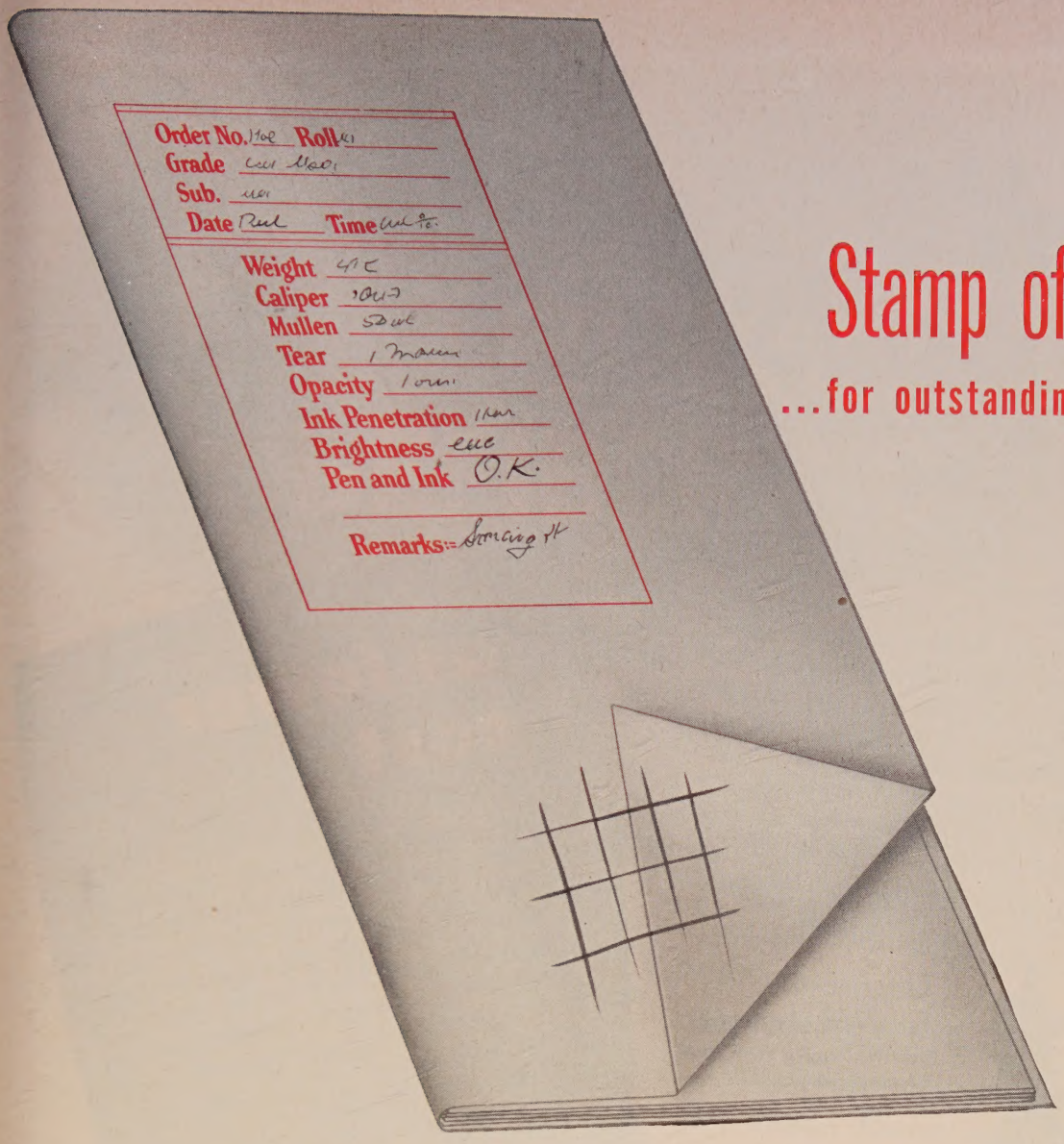

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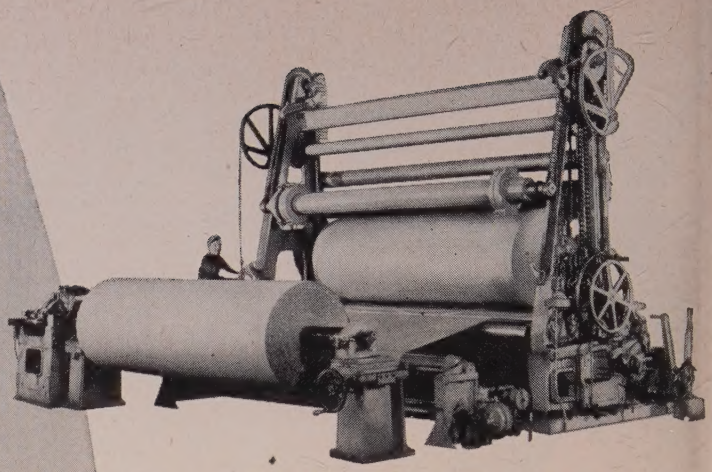

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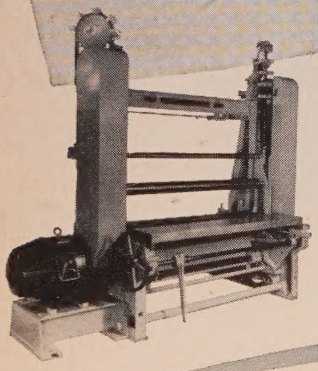

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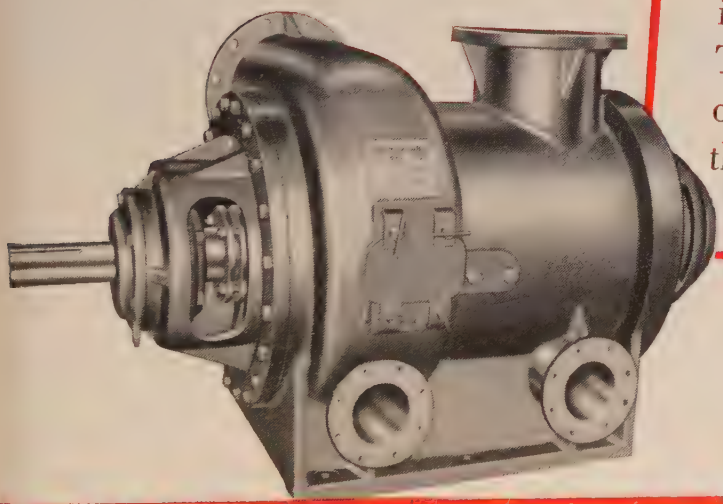

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PEOPLE HAVE A PLACE IN YOUR PLANNING

A. J. MILLER, JR.

You will note that my subject is "People Have a Place in Your Planning." The idea is certainly not a new one. I presume that this thought has been a subject of many, many talks at numerous meetings, and there is no doubt that a great many reams have been written about it. Yet, in spite of all this talk and all these words, the fact remains that little has been done about the problem. An eminent industrialist, Henry Ford, II, said, "Machines alone do not give us mass production. Mass production is achieved by both machines and men. And while we have gone a long way toward perfecting our mechanical operations, we have not successfully written into our equation whatever complex factors represent man, the human element." I agree with Mr. Ford.

Our society has been condemned both at home and abroad, for having made tremendous strides in material and scientific achievement, to the neglect of our human and spiritual progress. We have been accused of overemphasizing the importance of money, machines, and materials at the expense of man. And in these accusations the engineers have come in for a good share of the blame.

Yes, all these facts are too well known. They have been repeated to the point where we can hardly bear to hear them any longer. Their importance is not in dispute. The question has become rather "What shall we do about them?"

Here are some of the common questions:

1. Since some people work out successfully on certain jobs while others fail miserably, can we solve the problem of employee adjustment by better selection or; is our industrial environment simply bad or wrong, too specialized for too many people?

2. Another question: To what extent should the engineer go—in building worker's skill and power into a machine? What should he leave out? To what extent is the human equation capable of affecting quality and quantity of the product?

3. Or again: if the most efficient approach to manufacture is to break jobs down into their simplest components, have we found the best way of organizing these components for the satisfaction and best interests of the worker?

4. Fourth and final, but by no means the last of these questions: At what stage of development or planning for industrial manufacture should the human element be considered?

These are but a few of the many questions that could be developed around this subject. Now there are two fundamental ways to start doing something about them.

A. We can work toward the better adjustment of men to machines, or

B. We can adjust or rebuild our machine environment to better fit the needs and personalities of men.

Which is the best way? I don't know. A great deal of study and research is necessary before we can answer that question. In the meantime I believe we should work on both facets of the approach at the same time, simply because time itself does not permit us to delay. Besides, these two approaches to the problem are not in conflict, but should prove complementary, one to the other.

A long time ago I learned it was dangerous to talk to an engineer in an illogical manner; nevertheless, today I would like to start with point (B) before we come to point (A). That is, I would like to discuss the suggested approach of "adjusting or rebuilding our machine environment to fit the needs and personalities of men." Certainly the surface has hardly been scratched by the engineers in their design of machines and equipment in terms of the people who will operate or attend them. Rather the designer has consistently considered only the function and performance of the machine itself with little or no regard to its effect on human beings. Our now famous assembly lines are an outstanding example of this: for example, an auto mechanic hanging by heels to repair a car. That the engineers are not ignorant of these possibilities, however, is indicated by the remarks made by one of the country's famous industrial designers. When asked what products lend themselves best to industrial design he replied—and I quote—

"With very few exceptions like the ball, the wheel, and an occasional propeller, all things made by man can be improved in some way or another by good design. But it is a matter of degree. The best measure I know is people. The products which can be improved most by good design are products which are used or operated or seen by people. In other words, the more involved the product is with human beings, the more it needs good design. So why not use man as the starting point for all design, even to the extent of drawing human figures into the blueprints?

"It must constantly be borne in mind that the object being worked on is going to be ridden in, sat upon, looked at, talked into, activated, operated, or in some way used by people individually or en masse. If the point of contact between the product and people becomes a point of friction, then the designer has failed. If, on the other hand, people are made safer, more comfortable, more desirous of purchase, more efficient —or just plain happier—by contact with the product, then the designer has succeeded."

If coupled with this effort on the part of your de-

A. J. Miller, Jr., Asst. Director of Public Relations, The Mead Corp., Chillicothe, Ohio.

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signers, equal efforts are made to reduce monotony and mental fatigue of the machine paced and repetitive jobs, and measures are taken to introduce a sense of achievement or fulfillment into the employees' work, a great deal can be accomplished in the direction of our first objective. To mention only a few of the possibilities:

1. Rotate jobs among employees engaged in standardized tasks to reduce their monotonous and fatiguing effects. This is practiced to some extent in certain finishing room operations in paper mills today and probably has wider application in other areas of our industry.

2. Pay more attention to the content of individual jobs in terms of the time cycle so that they can be made to encompass a larger number of operations than usual. This is sometimes called "job enlargement."

3. Another idea that has great possibilities in terms of increasing worker satisfaction, is to provide more opportunities for team play and teamwork in the organization of jobs. It has been rather conclusively demonstrated that where the job situation permits or requires group interaction and group interdependence, that higher employee morale will result.

Of course, the degrees to which any of the above suggestions can succeed in given instances will vary, and they are in conflict with the somewhat prevalent belief, that the proper approach to the problem of human variability and human failure is to introduce *more* automatic machines and *more* foolproof processes to eliminate the influence of the human element. Without admitting the validity of this belief, it still will not solve the problem of employee unrest and employee dissatisfaction so prevalent in our industrial society of today. In fact, this unrest has largely been produced by the principle described, that is, the elimination or minimization of the human factor in production.

As a corollary to the long range objective of adjusting the industrial environment to fit the needs of men, we should reconsider the education of the engineer. I do

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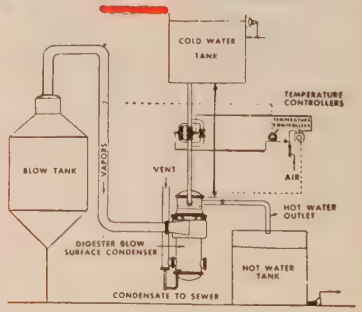

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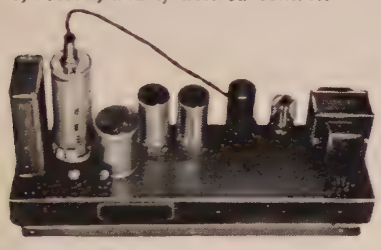
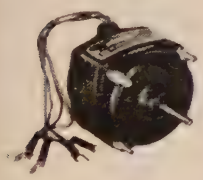


Good engineering shows in this Amplifier's thorough filtering, high impedance, and plug-in connection to the rest of the Speedomax.

Good engineering shows in this Slidewire's non-inductive winding and in absence of any flexible leads which might form inductive loops.



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not propose in these remarks to outline or suggest an educational program for engineers of the future, but I would like to make this point. We in the industrial relations field have taken engineering graduates and have given them additional training to make personnel managers and industrial relations executives out of them. How many personnel managers and industrial relations people have you made into engineers?

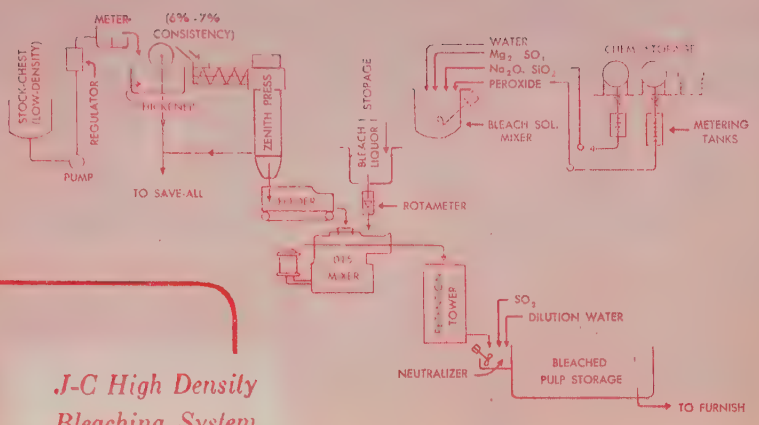
Let's go back now to point (A), our second approach to the problem of incorporating people in our planning. That is "the better adjustment of men to machines."

This approach accepts the inevitability of change in our industrial system. We all know we will continue to develop new tools, machines, and equipment designed to improve the quality and quantity of production, and to eliminate or reduce expensive labor costs. The effect of these changes upon the worker are twofold. They may require him to learn new skills, new ways of doing a job to which he has become accustomed through a lifetime of experience, or to surrender some portion of his skill to the mechanical or electronic skill of the machine. Sometimes they will absorb his job altogether forcing him to find another job within the plant or in the community. This second effect, which in simple terms means insecurity for the worker, is the one which is perhaps the most disturbing. In the paper industry we are fortunate in that most of the unions representing pulp and paper mill workers have accepted the principle that manufacturing changes are not only inevitable but in most cases desirable. The unions admit the right of management to introduce new or improved production methods, reserving, of course, their right to bargain over the results of these changes as they affect the employee's pay, his job security, and his seniority. In this favorable environment what can we do to prepare for the impact upon people—to cushion the effect of engineering changes upon the worker—as these changes come from your drawing boards and develop into reality?

First, we can improve the selection of employees so that insofar as possible employees will be psychologically and temperamentally suited to the kinds of work they can do best. We recognized at the outset that there are individual differences in people—that some will succeed on certain jobs and others will fail. But, if selection is to be a partial answer to the problem, it will require planning and this planning must take place in advance of the time when the selection of the employees is to be made. The development of selection methods and techniques requires time and painstaking research, therefore, plans for better selection must be integrated with engineering plans for better production, long before the latter become a fact in the plant.

Second, *we can* train people in new skills and abilities which will broaden their usefulness, make them more flexible, and thus capable of adjusting to the requirements of an ever-changing industrial system. Great strides were made in training during the last war when, because of necessity, it had to be done and done quickly. However, we will not have capitalized fully upon that experience unless we coordinate our training plans with engineering planning while the engineering plans are being formulated. I am sure that many of you have had the experience of installing a new piece of equipment, designed to improve the quality or rate of

Jackson and Church HIGH DENSITY SYSTEM



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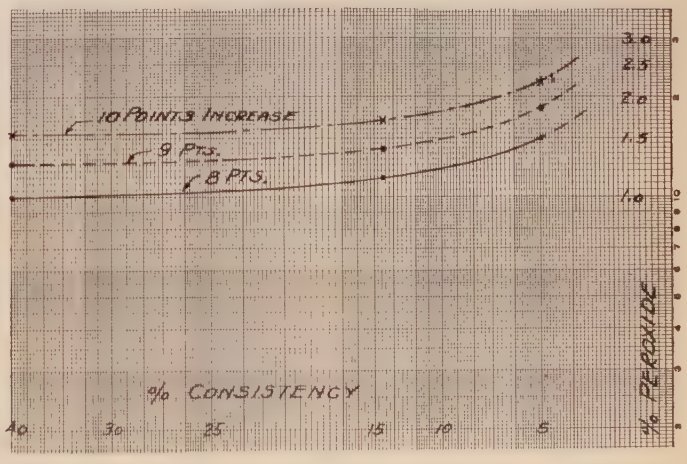

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production in your mill, only to find at the last minute that your hopes were not achieved or were long delayed because of human failures. The people just were not trained or prepared in advance to do the job you wanted. While these failures cannot be eliminated they can be drastically reduced if preplanning and pretraining are coordinated with engineering plans.

The third and most vital step that can be taken, is to assure proper coordination of planning in the initial stages of any engineering change. You should be certain to provide at the very beginning for participation of (1) those at the highest level in the organization, (2) those who will be responsible for manning the new plant or new piece of equipment, (3) and those who will be responsible for making the personnel changes which will result from the installation. This seems a simple solution, yet perhaps because of its very simplicity it is often not done. What I am talking about is to bring your industrial relations manager or personnel manager into the picture while your engineering plans are still in the talking stage. Don't wait until you are ready to turn the new machine over for the first time to ask him for a machine crew. Don't wait until a week or two before you have completed a new power unit to ask what to do with the people on the jobs you have eliminated or replaced. Make your personnel or industrial relations executive an integral part of your operations planning committee (by whatever name it may be called) so that he will be constantly aware of what is going on. Then he can see to it that man-power planning proceeds in the same sort of logical orderliness that you expect from the work of the designer, the draftsman, the purchasing agent, the supplier, and the builder of the equipment. Properly informed, your industrial relations people can follow a blueprint too, so that men will be selected and trained for the anticipated date of operation, so that wage and salary administration can plan for rate and salary changes, and so that labor relations can try to negotiate a labor agreement with sufficient flexibility to handle your man-power problems when they occur.

Perhaps the most important by-product of this type of planning will be the ability of management to inform the people who will be involved in a proposed change of just how it will affect them, and informed of it in advance. There is perhaps nothing more damaging to employee morale than for them to see changes developing around them and about them with little or no understanding of what these changes will do to them. We all like to hold on to what we have—whether this be money, a job, or seniority. If we see these things being threatened, imagination, fired by the rumor factory, can offset the advantages expected from the improvement we are planning to make.

Perhaps these remarks do not appear to be constructive. I have certainly not tried to answer all of the problems wrapped up in the question of people and their place in industry. I have tried in the time allowed to suggest for your consideration some of the avenues by means of which we can approach certain of these problems. Nor are the suggestions I have made new or novel. They are familiar as ways of solving many of your engineering and manufacturing difficulties.

While we cannot solve our problems with people with the same simplicity or with the same degree of standardization that we can mix concrete or harden steel, we can adopt an organized system of planning for the problems of the people in industry.

All of this will require time and effort, and it may appear that such concern for the problems of people in your planning will be too much for an already overloaded excutive or engineer. I believe this difficulty is more apparent than real. If you don't make these plans before hand, it is my belief that you will spend twice as much time trying to figure your way out of the problems that you will create. In considering whether the time and effort are worth the expected results, reflect for a moment upon the great amount of energy and time that is normally devoted to the analysis of machines and equipment, markets and product that precedes the decision to make a new product or to build a new paper machine. Perhaps the human resources of your organization merit some such similar degree of attention in your plans.

PRESENTED at the Engineering Division Luncheon, at the Seventh Engineering Conference of the Technical Association of the Pulp and Paper Industry, Chicago, Ill., Oct. 13–16, 1952.

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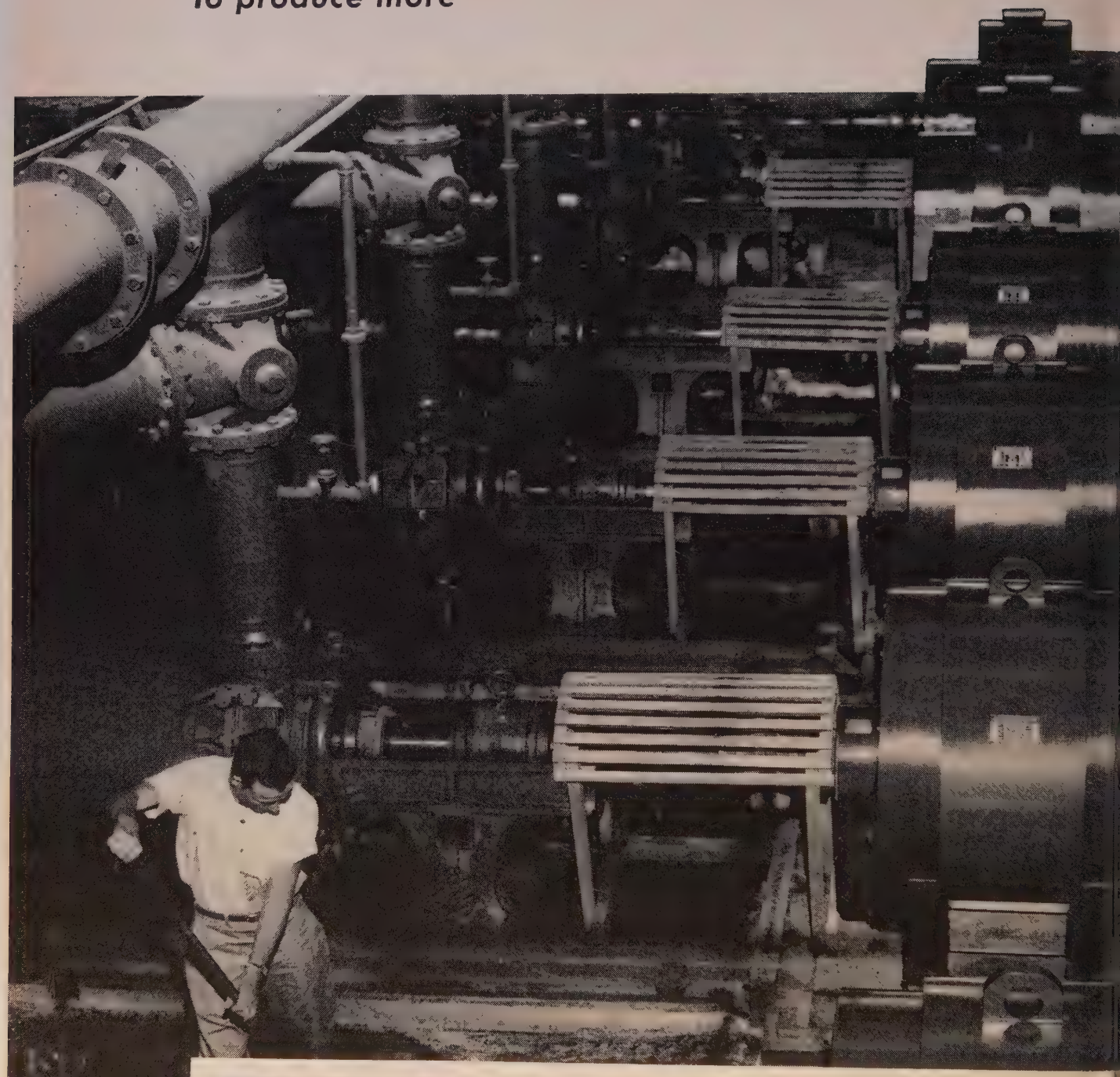

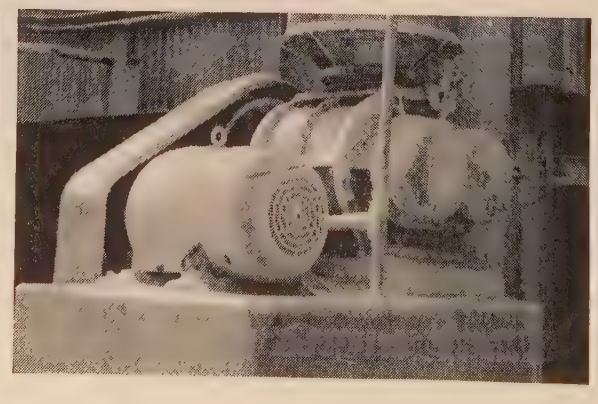

Westinghouse Life-Line motors drive Hudson's Fuller Lime Unloaders and they're constantly covered with lime. When asked about trouble, their electrical foreman replied, "Trouble? You would never know they're on the place."

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all the attention needed. Several open spaces in th
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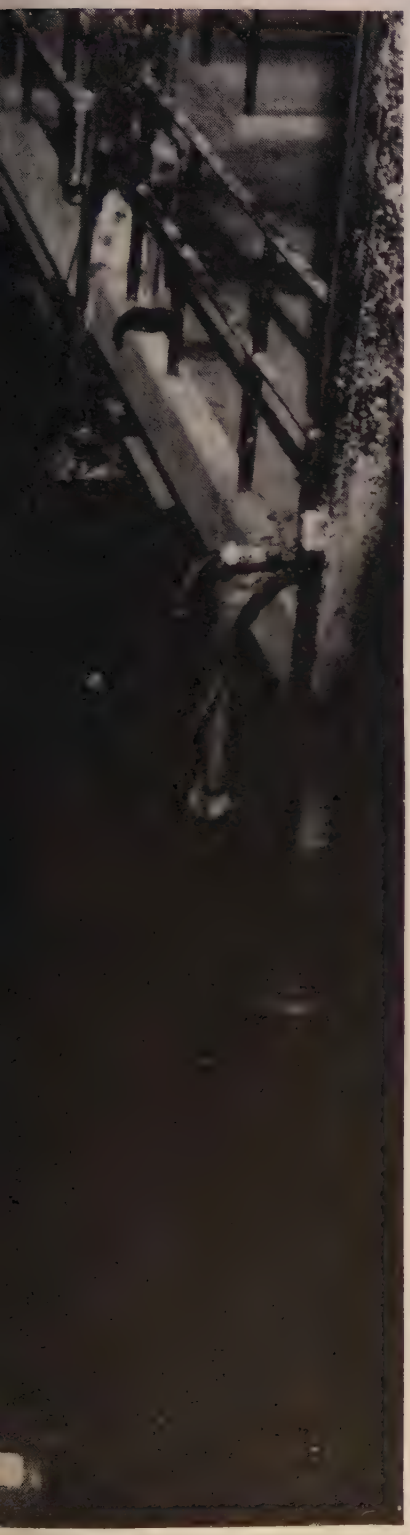
How the right electrical equipment reduces costs at Hudson Pulp & Paper



leven Westinghouse synchronous motors rive the Jordans and provide the abso- utely constant speed needed for uniform brillation. Westinghouse synchronous notors also help produce the highest nch-cut potential on the Victory Beaters not shown).

The right electrical equipment for stock preparation and other processes can play an important part in boosting paper mill production. You can get more continuous operation and reduced maintenance. There are many good examples in the newly expanded mill of Hudson Pulp & Paper Company at Palatka, Fla.

There's no chance of a log conveyor jam-up because Westinghouse controls on all conveyors to the barker are specially interlocked. If a stoppage occurs in one section, the whole conveyor system will automatically stop until the trouble is located and cleared.

They get a well-balanced steam-electric load by using two Westinghouse turbines and one turbine-motor combination for driving feed water pumps. Under normal conditions, the turbines drive the pumps and the motor is idle. But if they need additional process steam, they can divert steam from the one turbine-motor combination by cutting out the turbine and cutting in the motor.

They have a more productive chipper operation as a result of installing special Westinghouse chipper motors. These motors are designed especially for this rugged service and they're built in a heavy, reinforced construction. They have been absolutely trouble-free.

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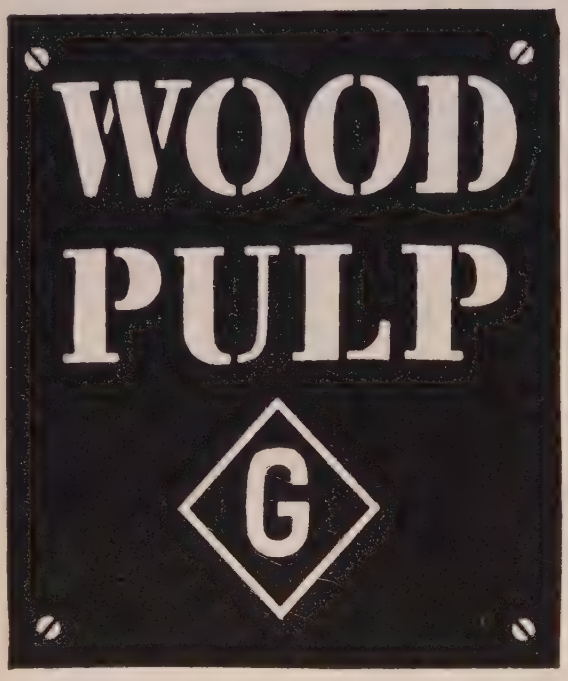

"In the full tide of successful experiment."
THOMAS JEFFERSON

One of the most important attributes of American pulp and paper makers is their strong devotion to research and experiment for developing new products and uses.

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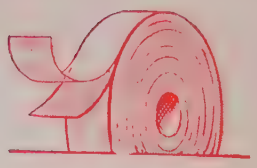

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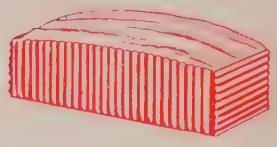
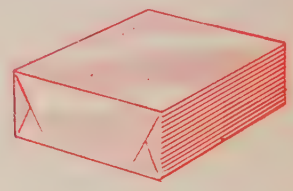
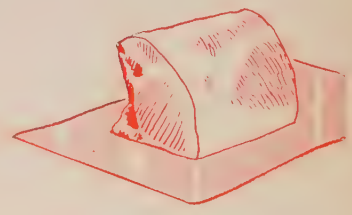


For bakery tray and pan liners, "Quilon" greatly reduces sticking of baked goods.



For frozen-food packages and locker wrap, "Quilon" eliminates fiber pull when frozen-food packages are separated and when frozen food is removed from wrap.



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THE ROLE OF LIFE INSURANCE COMPANIES IN PAPER INDUSTRY FINANCING 1949–51

HOWARD BAUMGARTEN

Life insurance companies are an important source of capital funds in the economy generally and for the paper industry specifically. Twenty of the 25 largest life insurance companies in the United States hold almost half a billion dollars worth of pulp and paper industry securities in their portfolios. Over 90% of this total is debt securities and amounts to $432 million. The balance consists of $33 million of equity securities—both preferred and common stock. The holdings of these 20 companies—both debt and equity securities—have increased more than four times the amount held in 1945. The largest holders of paper industry securities are Prudential with $114 million and Metropolitan Life with $89 million. The changes that occurred from year to year have been due to the investment operations of a small number of these insurance companies. The investment activities of New York Life and Prudential have been most important during the past three years. Seventeen of the 20 insurance companies invested in preferred stock and only 5 held common stock in the pulp and paper industry. This study shows the continued role of insurance companies in providing funds for expansion in the years since the previous study was made in 1949.

In 1949 Dr. Shallcross made a study of the role life insurance companies were playing in the postwar expansion period.* That study was based on questionnaires sent to the 25 largest life insurance companies in the United States. Five of the companies replied in 1949 that they did not hold any securities in the pulp and paper industries.† The remaining 20 companies provided the data on which her study was based. In order that the figures gathered in this survey would be directly comparable, the 1952 questionnaire was sent to the same companies.

On Dec. 31, 1951, the combined assets of these 20 companies were $51 billion which is 70.5% of the combined assets of all life insurance companies in the United States. Although this is a large majority of the insurance money available for investment, we do not mean to infer that this is a statistical sample designed to give estimates of the total magnitude of paper industry investments. Many small insurance companies which are located in pulp and paper producing areas may have substantial investments in the industry.

HOWARD BAUMGARTEN, Research Assistant, The Institute of Paper Chemistry, Appleton, Wis.

* "The Role of Life Insurance Companies in Paper Industry Financing in the Postwar Period—1945–49" by Ruth E. Shallcross. *Paper Trade J.* (April 20, 1950).

† Although "pulp and paper companies" are defined as only primary producers, some questionnaires included small amounts of "converters" securities. We have not changed any reported figures.

SUMMARY OF THE 1949 STUDY

In 1948 20 of the 25 largest life insurance companies held over $250 million worth of pulp and paper industry securities.

About 90% of this total was debt securities and amounted to $250 million. The balance consisted of $28 million of preferred and common stocks.

The largest holders of paper industry securities were Metropolitan Life with $55 million and Northwestern Mutual with $44 million.

Of the 25 companies, 17 held preferred stocks of the paper industry and 5 held common stocks.

The 20 life insurance companies expanded their holdings over 2.5 times in the period 1945–48. An increasing proportion of their assets was invested in railroads, public utilities, and manufacturing industries.

Paper industry assets increased $1.8 billion (60%) from 1945's $2.9 billion to 1948's $4.7 billion. This represented an increase in capacity of four million tons.

From an extensive list of specific important paper industry financing the needed capital was obtained as follows: 13.8% from common stock, 12.0% from preferred stock, and 74.2% from debt financing.

Of the $1.8 billion increase in paper industry assets, approximately $550 million was raised by indebtedness and 50 to 60% of this amount was obtained from insurance companies.

INSURANCE COMPANY INVESTMENT IN THE PAPER INDUSTRY

In 1951, the amount of paper industry securities held by 20 large life insurance companies was approaching the half billion dollar mark** (see Table I). In 1945 these companies held $106 million; at the end of 1951 the total was $465 million. This is more than a four-fold increase.

Breaking down this $465 million total, debt securities amount to $432 million (see Table II) and equity securities amount to $33 million in 1951 (see Table III).

From Table II, it is seen that the holdings of debt securities have increased from $98 million in 1945 to $432 million in 1951. This is a 4.39-fold increase in the 6-yr. period 1945–51. The growth of debt holdings reflects the rapid industrial growth and the inflation of the postwar period. Of course, debt securities have been the traditional instrument for insurance company investment. Two additional reasons for this large

** Throughout this paper, in order to facilitate reading, all figures will be given in round numbers. Exact values are given in the tables.

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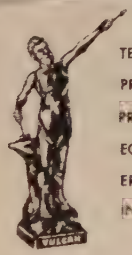

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Table I. Paper Industry Securities Held by 20 Major Life Insurance Companies

1945–51 ($000 Omitted)

	1951	*1950*	*1949*	*1948*	*1947*	*1946*	*1945*
Total	$464,899	$368,634	$313,428	$277,963	$197,586	$161,570	$106,072
Increase over previous year	96,265	55,206	35,465	80,377	36,016	55,498	....

Source: Questionnaires from insurance companies.

growth are the postwar tax structure and the relative cheapness of bond interest rates, both of which favor debt over equity financing.

In Table IV are listed the larger changes in holdings of debt securities during 1951 and during 1950. A relatively few companies dominate the trend. During 1951, the Equitable Life Assurance's increase of $23.5 million and Prudential's increase of $57 million were by far the largest changes. These two figures total $80.5 million and account for 82% of the $98 million increase in debt security holdings that year.

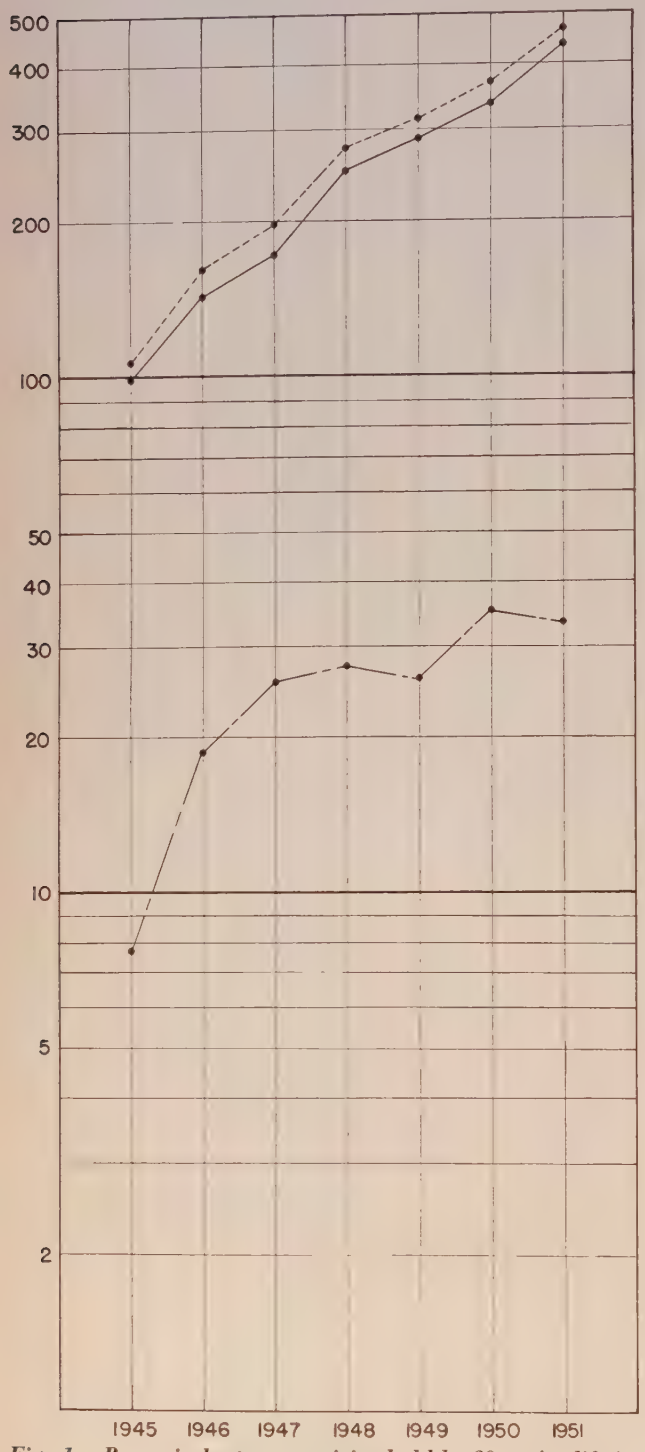

Fig. 1. Paper industry securities held by 20 major life insurance companies

– – – – Total securities
——— Bonds
–·–·–·– Stocks

During 1950, 93% of the increase was accounted for by Metropolitan Life's $8.7 million, New York Life's $14 million, and Prudential's $20 million increase in debt security investments.

Turning to Table III and an analysis of the preferred and common stock holdings of the 20 life insurance companies, the pattern does not significantly differ from the pattern of debt holdings. Equity holdings have increased from $8 to $33 million in the 1945–51 period. This is a 4.37-fold increase, as compared with a 4.39-fold increase for the debt securities.

Breaking down the changes in Table V a few companies again have dominated the trend. During 1951 Northwestern Mutual and Mutual Benefit, New Jersey, each decreased their holdings of equity securities by over $1.5 million and Mutual Life, New York, by 0.5 million. This decrease of $3.5 million was only partially offset by Prudential's increase of $2.9 million. The net difference (−$742,000) is slightly less than 50% of the 1951 decrease in equity holdings of $1.6 million.

During 1950 the three large increases (John Hancock's, New England Mutual's, and Prudential's holdings, amounting to $8.6 million) accounted for 90% of the increase for the 20 companies.

In analyzing both tables, the changes are fairly well scattered among the 20 companies. One exception is Prudential which has registered a significant increase in its paper industry holdings each year.

We in the paper industry always think in terms of the growth characteristics of the industry. A growing industry is always in need of new capital. In Figs. 1 and 2 are the data of Tables I, II, and III, presented on semilogarithmic scale paper. Since equal vertical distances are equal percentages of growth, this allows the comparison of the two series of data. In Fig. 1 it will be noted that the rate of growth in equity holdings was very rapid in the 1945–47 period when several state laws were changed so that life insurance companies might invest in equity securities. Since 1947 there has been a leveling off, with slight decreases in 1948 and 1950.§

The curve of debt securities is almost a straight line function for the 1945–51 period. Since the debt holdings are about 10 times as great as the equity holdings, the total securities curve is very similar to the debt holdings curve.

In Fig. 2 we have superimposed the two curves—debt and equity holdings to draw attention to the difference in rate of change between the two. As insurance com-

§ It should be noted that a trend should not be calculated from these data. The period involved is far too short to have any long-term significance.

Human papermakers learned well from the wasp

As early as 1719, the celebrated naturalist, René Réamur reported the manner in which the wasp made paper from wood . . . where man used only rags and linen. Observing the world's first professional papermaker in action, Réamur learned that the wasp chews wood fibers, mixes them with a natural size and spreads this paste to dry into a paper substance for its nest.

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SIZING STARCHES
Staley makes a full range of starches and dextrins for beater, surface or calender sizing.

CORRUGATING AND LAMINATING
Choose Staley's finer corrugating starches, special laminating and bag adhesives.

COATING ADHESIVES
Staley Starches and Dextrins give better results for machine or regular brush coating.

PLASTICIZERS
"Sweetose" and Staley's Corn Syrup are ideal softening agents for glassine paper

Table II. Paper Industry Debt Held by 20 Major Life Insurance Companies

1945–51 ($000 Omitted)

	1951	1950	1949	1948	1947	1946	1945
Aetna	$ 6,936	$ 6,258	$ 4,357				
Bankers	1,050	804	810				
Connecticut Mutual	2,313	1,503	1,279				
Equitable Life Assurance	61,807	38,247	37,840				
Equitable Life, Iowa	1,687	1,164	1,059				
Guardian	248	247	255				
John Hancock	26,255	25,688	27,358				
Home Life	1,394	901	921				
Lincoln	308	338	369				
Massachusetts Mutual	20,840	21,624	21,785				
Metropolitan Life	84,863	83,937	75,267				
Mutual Benefit, N.J.	6,410	3,330	3,500				
Mutual Life, N. Y.	18,753	16,312	14,294				
New England Mutual	5,687	5,856	6,701				
New York Life	35,713	29,188	15,228				
Northwestern Mutual	36,830	34,885	32,887				
Penn Mutual	9,899	11,420	11,502				
Phoenix	2,243	1,069	1,117				
Provident Mutual	2,323	1,974	2,150				
Prudential	106,535	49,442	29,222				
	$432,094	$334,187	$287,901	$250,072	$171,830	$142,687	$98,421

Source: Questionnaires from insurance companies.

panies approach the legal limits or when the stock market is in historically high-price territory, we may expect the equity investments to grow less rapidly.

In addition, the questionnaire requested the 20 companies to calculate what percentage paper industry securities were of (1) total company assets, (2) total company corporate securities,‖ and (3) total company industrial securities.¶

Table VI indicates that the average of the 20 companies had just over 0.6% of their total assets invested in the paper industry. The percentage varied from 0.14% for Guardian Life and 0.15% for Lincoln National to Massachusetts Mutual's 1.5%. Massachusetts Mutual's holdings, although the highest in percentage terms, were slightly below average in dollar terms (see Tables II and III). Aetna's holdings were small in both percentage and absolute terms.

‖ "Corporate securities" includes securities of railroads, public utilities, and total industrial corporations.

¶ "Industrial securities" includes securities of all private corporations including banks, insurance companies, and other service or financial organizations but excludes railroads and public utilities.

Of course, a large insurance company with a small percentage of paper industry securities will far outweigh small insurance companies with large percentages of their assets in paper securities. As an example, New York Life's holdings approximate $37 million but are only 0.72% of total assets, a percentage not too far from the average.

The arrows in Table VI indicate recognizable trends for the years 1949, 1950, and 1951. Four companies have an upward trend in the percentage of total assets invested in the industry; four companies have a downward trend; the remainder show no trend.

Table VII shows that pulp and paper securities are an average 1.41% of total corporate securities held by the 20 companies. The extremes are Lincoln National's 0.5% and Prudential's 3.7%.

The most interesting and important information in this area is shown in Table VIII where we have an indication of what portion of the investment portfolio is attracted to the paper industry as compared with other manufacturing industries. In this classification rail-

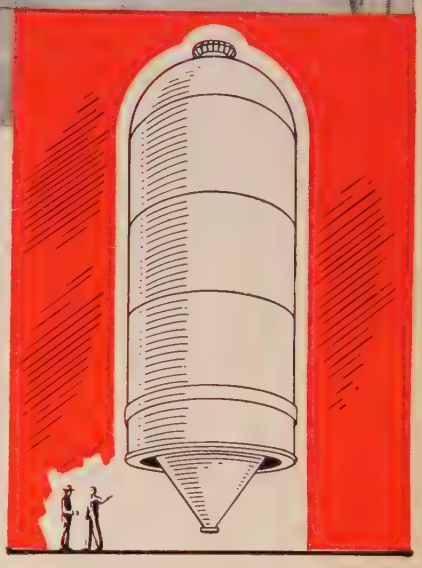
Table III. Paper Industry Equities Held by 20 Major Life Insurance Companies

1945–51 ($000 Omitted)

	1951	1950	1949	1948	1947	1946	1945
Aetna	$ 1,128	$ 1,224	$ 419				
Bankers	183	183	287				
Connecticut Mutual	1,152	1,510	1,518				
Equitable Life Assurance	0	0	0				
Equitable Life, Iowa	534	540	647				
Guardian	210	220	230				
John Hancock	8,810	8,850	3,402				
Home Life	98	109	105				
Lincoln	443	359	512				
Massachusetts Mutual	1,332	1,365	1,307				
Metropolitan Life	3,886	3,954	3,978				
Mutual Benefit, N. J.	192	1,700	1,700				
Mutual Life, N. Y.	632	1,138	1,144				
New England Mutual	3,521	3,998	2,617				
New York Life	1,137	1,222	1,103				
Northwestern Mutual	1,842	3,463	3,771				
Penn Mutual	702	762	747				
Phoenix	0	0	0				
Provident Mutual	0	0	0				
Prudential	7,013	4,120	2,040				
	$32,805	$34,447	$25,527	$27,891	$25,756	$18,883	$7,651

Source: Questionnaires from insurance companies.

140 tons of chips per cook!



When welded into a complete unit this became the world's largest lined sulphite digester. 58 feet in height, the inside diameter is 19 feet, and it will take about 140 tons of chips per cook. The inside is Smithlined, bonded to the carbon steel digester walls by A. O. Smith's time-proven process of closely controlled resistance spot welding.

That's the capacity of Rayonier's new Smithlined Sulphite digester installed at their Fernandina, Florida Division

Important news to pulp and paper men, because it means more pulp production per day.

Important news to paper men also, to know that A. O. Smith had the design engineering, manufacturing know-how, and plant facilities to successfully handle this huge digester from blueprint to finished installation.

HERE'S HOW WE DID IT!

1. Our metallurgical and welding engineers, who have been working with Rayonier for a number of years on the use of corrosion resistant linings for digesters, collaborated with their engineers on the selection of the corrosion resistant linings and welding procedures.
2. Too big to be shipped in one piece, our design engineers planned the production for a maximum amount of shop assembly to hold expensive field construction to a minimum.
3. Furnished field welding specifications that produced a digester of the same rigid high quality standards as those manufactured under the exacting controls of A. O. Smith shop practice.
4. Provided a welding engineer with over 20 years' experience to control the execution of the specifications, hire, qualify, and supervise all welders and personally handle and evaluate all test procedures.

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Los Angeles 22 • Midland 5, Texas • New Orleans 12 • New York 17 • Pittsburgh 19 • San Francisco 4 • Seattle 1
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International Division: Milwaukee 1

Table IV. Major Changes in Holdings of Debt Securities
1949–50 and 1950–51 ($000 Omitted)

	1950–51	1949–50
Aetna	...	+$ 1,901
Connecticut Mutual	+$ 810	...
Equitable Life Assurance	+ 23,560	...
Metropolitan Life	...	+ 8,670
Mutual Benefit, N. J.	+ 3,080	...
Mutual Life, N. Y.	+ 2,441	+ 2,018
New York Life	+ 6,525	+ 13,960
Northwestern Mutual	+ 1,945	+ 1,998
Penn Mutual	− 1,521	...
Phoenix	+ 1,174	...
Prudential	+ 57,093	+ 20,220
	+$95,107	+$48,767

Table V. Major Changes in Equity Security Holdings
1949–50 and 1950–51 ($000 Omitted)

	1950–51	1949–50
John Hancock	...	+$5,178
Mutual Benefit, N. J.	−$1,508	...
Mutual Life, N. Y.	− 506	...
New England Mutual	...	+ 1,381
Northwestern Mutual	− 1,621	...
Prudential	+ 2,893	+ 2,080
	−$ 742	+$8,639

Source: Questionnaires from insurance companies.

road and public utility securities, which traditionally take a large portion of insurance money, are eliminated. The average for the 20 companies is 4%. In 1951 this percentage varied from Guardian's 1.4 to Northwestern's and Phoenix's 10.

There are several noticeable trends in this table. Nine of the companies have decreased the percentage of their industrial portfolio invested in the paper industry since the last survey was made. Some of these downward trends extend unbroken from 1944. Only four companies have an uptrend in holdings in this classification since the last survey. They are Equiable Life, Iowa, Home Life, New York Life, and Prudential. Seven companies show no perceptible trend.

Two outstanding examples of the predominant downtrend are: Northwestern Mutual's holdings fell from 26% in 1945 to 10% in 1951. John Hancock's holdings fell from 13.5% in 1946 to 5.5% in 1951. In dollar terms this trend is not noticeable, since total funds to be invested have risen sharply in this period The smaller percentages of 1951 apply to larger total assets.

PAPER INDUSTRY REQUIREMENTS

We have been investigating the life insurance company side of the capital expansion program. Economists would call it the "supply" side of the picture. Now let us briefly turn to the paper industry or demand aspects of the picture. Column 1, Table IX, shows the amounts spent each year by the paper and allied industry for new plant and equipment, 1945 through

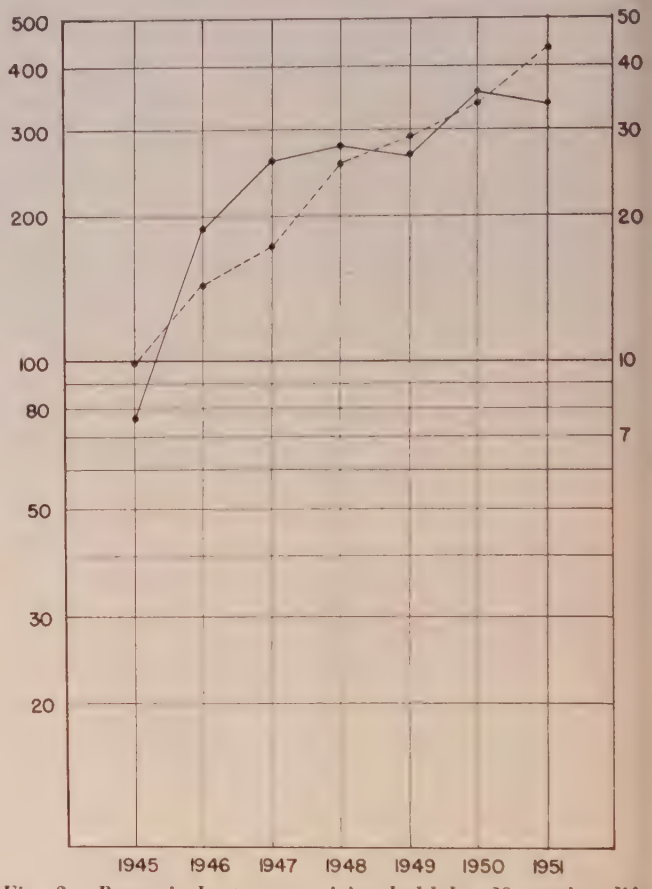

Fig. 2. Paper industry securities held by 20 major life insurance companies

– – – – Paper industry debt (left scale)
——— Paper industry equity (right scale)

Table VI. Pulp and Paper Securities as a Percentage of Total Assets

	1951	1950	1949	1948	1947	1946	1945
Aetna	0.41	0.41	0.29	0.32	0.26	0.21	0.23
Bankers	0.21	0.18	0.22	0.25	0.10	0.07	0.02
Connecticut Mutual	0.39	0.36	0.36	0.36	0.25	0.22	0.08
Equitable Life Assurance	↑1.01	0.67	0.72	0.45	0.22	0.27	0.39
Equitable Life, Iowa	0.49	0.40	0.43	0.33	0.36	0.33	0.60
Guardian	↓0.14	0.16	0.17	0.19	0.21	0.11	0.12
John Hancock	1.08	1.16	1.14	1.06	1.15	1.11	0.06
Home Life	0.59	0.43	0.46	0.49	0.53	0.52	0.31
Lincoln	↓0.15	0.15	0.21	0.23	0.27	0.38	0.24
Massachusetts Mutual	↓1.51	1.65	1.76	2.33	2.12	1.85	1.40
Metropolitan Life	↑0.8	0.9	0.8	0.60	0.45	0.21	0.10
Mutual Benefit, N. J.	0.50	0.40	0.42	0.44	0.02	..	..
Mutual Life, N. Y.	0.88	0.79	0.74	0.80	0.60	0.90	0.80
New England Mutual	0.73	0.84	0.88	0.71	0.66	0.60	0.16
New York Life	↑0.72	0.62	0.34	0.38	0.33	0.27	0.26
Northwestern Mutual	↓1.4	1.5	1.5	1.8	1.6	1.7	1.2
Penn Mutual	0.79	0.94	0.99	0.77	0.59	0.61	..
Phoenix	0.39	0.20	0.22	0.14	0.15	0.16	..
Provident Mutual	0.34	0.30	0.35	0.30	0.14	0.09	..
Prudential	↑1.19	0.60	0.38	0.42	0.14	0.06	0.04
	Average 0.63%	0.58%	0.59%				

Source: Questionnaires from insurance companies

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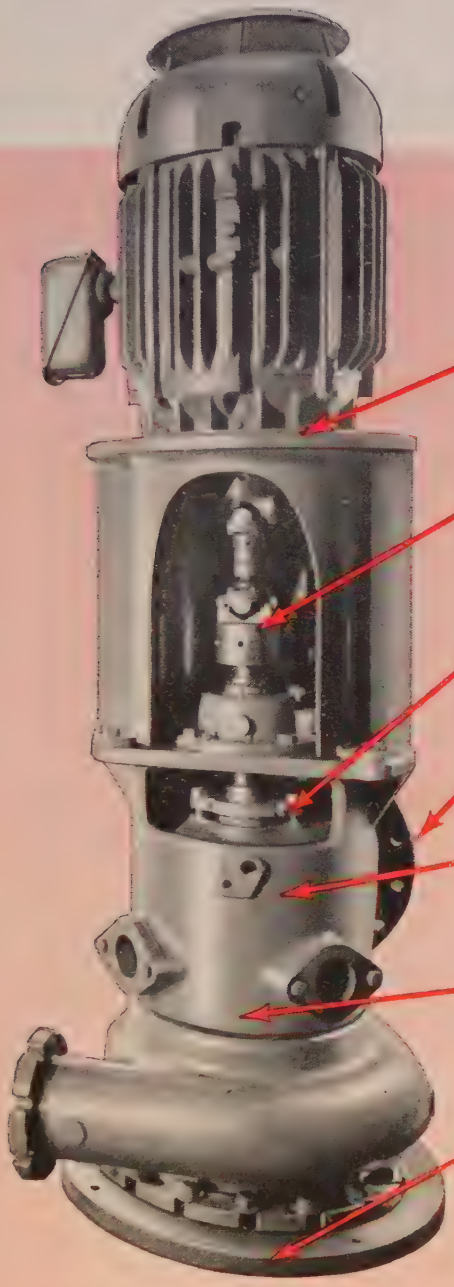
with ALLIS-CHALMERS PAPER STOCK PUMP



NO ALIGNMENT

Motor is bolted directly to motor pedestal, connected through universal joint spacer coupling. No close alignment necessary.

ACCESSIBLE COUPLING

Open motor pedestal allows easy access to universal joint type coupling, yet maintains rigid alignment.

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Stuffing box is easy to get at, too, making inspection and proper maintenance simple.

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16-inch suction opening on all pump sizes means low velocity at suction. Most air binding problems are eliminated.

GRAVITY FEED

Stock feed into eye of impeller is accelerated by gravity. Heavier stocks can be handled without difficulty.

LESS SPACE

Vertical design requires only one quarter as much floor space as a horizontal pump of the same capacity.

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Foundation is smaller and simpler than with horizontal design. Foundation costs are reduced.

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Table VII. Pulp and Paper Securities as a Percentage of Total Corporate Securities

	1951	1950	1949	1948	1947	1946	1945
Aetna	0.80	0.84	0.64	0.82	0.81	0.74	0.78
Bankers	0.66	0.66	0.79	1.07	0.62	0.54	0.18
Connecticut Mutual	0.75	0.67	0.65	0.87	0.65	0.57	0.20
Equitable Life Assurance	1.77	1.17	1.25	0.77	0.46	0.70	1.40
Equitable Life, Iowa	1.58	1.34	1.39	1.08	1.34	1.42	0.33
Guardian	↓0.54	0.69	0.73	0.85	1.14	0.70	0.74
John Hancock	1.93	2.15	2.18	2.09	2.49	2.57	1.42
Home Life	2.18	1.60	1.68	1.73	1.96	2.10	1.38
Lincoln	↓0.51	0.53	0.77	0.99	1.21	1.86	1.68
Massachusetts	↓2.77	2.97	3.06	3.92	3.97	3.80	2.94
Metropolitan Life	1.70	1.90	1.90	1.52	1.39	0.87	0.44
Mutual Benefit, N. J.	1.28	1.01	1.07	1.17	0.05	..	..
Mutual Life, N. Y.	1.73	1.62	1.57	1.80	1.60	2.80	3.10
New England Mutual	1.31	1.42	1.47	1.23	1.23	1.24	0.34
New York Life	↑1.53	1.35	0.83	..	..	..	..
Northwestern Mutual	2.00	2.00	1.90	3.60	3.70	4.00	3.20
Penn Mutual	1.56	1.94	2.02	1.61	1.35	1.59	..
Phoenix	1.58	0.81	0.94	0.73	0.87	0.96	..
Provident Mutual	0.69	0.63	0.74	0.66	0.40	0.28	..
Prudential	↑3.69	2.05	1.36	1.64	0.66	0.32	0.24
Average	1.41%	1.26%	1.28%				

Source: Questionnaires from insurance companies.

1951. Over $2 billion have been spent during the period.

Column 4 of Table IX relates the amount spent each year by the industry to the increase in security holdings of the 20 insurance companies. This varies from 10% in 1947 to over 20% in 1946, 1948, and 1951. If we were to survey all life insurance companies, the average percentage of expenditures for plant and equipment obtained from insurance companies might well be approximately 25%. That is certainly an important and significant source of funds. If further we were to subtract the undistributed corporate income from the expenditures for new plant for the years 1949 through 1951, the importance of the insurance company investment is more evident. Of the $1119 million spent for new plant and equipment, $888 million was financed by retained income. The remainder, $231 million, compares with the increase in insurance company investments of $187 million in the period.

Table VIII. Pulp and Paper Securities as a Percentage of Industrial Securities

	1951	1950	1949	1948	1947	1946	1945
Aetna	3.07	4.50	3.69	4.80	4.50	4.20	5.60
Bankers	↓2.47	2.78	3.17	4.46	2.67	2.97	1.20
Connecticut Mutual	↓4.25	4.51	4.67	4.90	3.86	3.38	1.46
Equitable Life Assurance	3.24	2.24	2.28	1.38	0.82	1.37	4.44
Equitable Life, Iowa	↑5.48	4.75	4.54	3.43	3.81	4.90	1.63
Guardian	↓1.35	1.88	2.04	2.90	3.50	2.10	2.70
John Hancock	↓5.57	6.81	8.02	10.08	12.35	13.65	9.55
Home Life	↑7.25	5.83	5.70	5.91	6.85	8.56	6.21
Lincoln	↓1.89	2.13	3.39	0.41	5.58	9.10	7.25
Massachusetts	↓6.58	7.28	7.15	9.23	10.63	13.94	13.74
Metropolitan Life	2.80	3.40	3.40	2.89	3.14	2.23	1.69
Mutual Benefit, N. J.	4.02	3.48	3.82	5.15	0.30	..	..
Mutual Life, N. Y.	3.37	3.20	3.22	3.80	3.50	6.30	8.30
New England Mutual	↓3.28	3.65	4.16	4.75	5.43	6.14	1.79
New York Life	↑3.80	3.62	2.32	5.24	6.84	7.58	13.23
Northwestern Mutual	↓10.00	12.10	13.30	16.50	16.6	21.00	26.00
Penn Mutual	↓3.66	4.68	4.85	3.92	3.91	5.26	..
Phoenix	10.09	5.92	7.60	6.30	6.77	8.85	..
Provident Mutual	1.90	1.86	2.33	2.27	1.53	1.24	..
Prudential	↑5.79	3.40	2.29	2.98	1.45	0.97	1.03
Average	4.49%	4.40%	4.60%				

Table IX. Manufacturer's Expenditures for New Plant and Equipment

1945–51 ($000,000 Omitted)

Year	Paper and allied industry[a]	Cumulative	Undistributed paper industry corporate income[b]	Yearly increase in insurance company paper industry holdings as a % of column 1
1945	$116	...	$ 72	..
1946	232	$ 348	254	24
1947	371	719	441	10
1948	383	1102	334	21
1949	298	1400	197	12
1950	327	1727	368	17
1951	494	2221	323	20

Source: [a] *Survey of Current Business*, December, 1951, Table 2, p. 17. [b] *Survey of Current Business*, July, 1952, Table 21, p. 21, and "National Income," 1951 ed., U. S. Dept. of Commerce.

CONCLUSION

Based on the survey, life insurance companies are an important source of capital funds. Paper industry assets have increased from $2.9 billion in 1945** to $6.6 billion in 1951, or 128%. In the same period, life insurance companies increased their investment in the paper industry by 340%. We must conclude that the insurance companies consider the paper industry a good investment.

** American Paper & Pulp Association, "Capital & Income Survey 1951."

Appreciation is expressed to Ruth E. Shallcross for her interest during the preparation of this paper.

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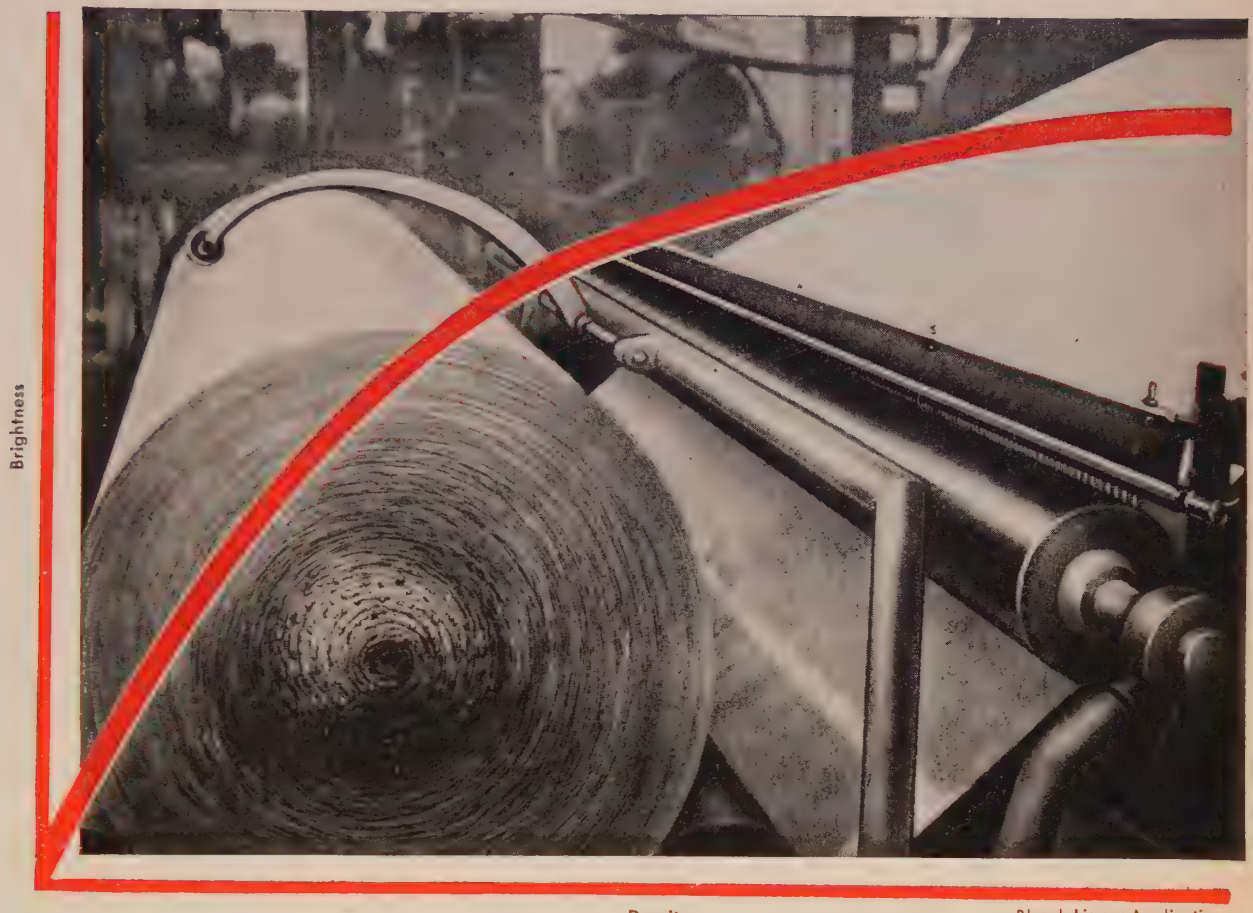

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Based on a series of trial runs, the operating conditions for the CHEMI-PULPER continuous digester were determined and the operation was placed on a 24-hour basis.

High yield kraft pulp in the 60-62% range was produced from a mixture of spruce and balsam woods. The pulp passed through hot stock refiners—over a vacuum washer—through the board machine disc refiners and onto the liner board machine. The liner board was made from 100% CHEMI-PULPER continuous digester kraft pulp at the usual mill operating speed and tested somewhat higher than the minimum requirements for .016 liner.

The CHEMI-PULPER continuous digester can produce kraft pulp continuously at a considerable *saving in steam and manpower* requirements, and at a *better uniformity* which is an inherent characteristic of a continuous process as compared to a batch operation.

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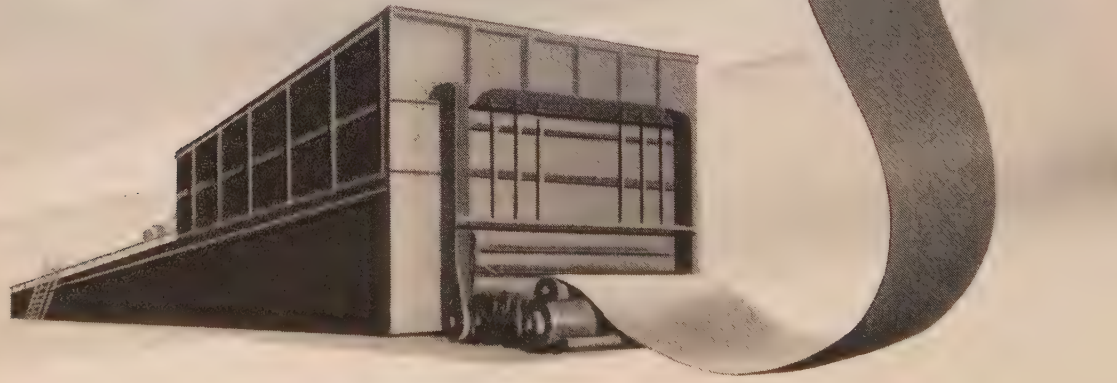

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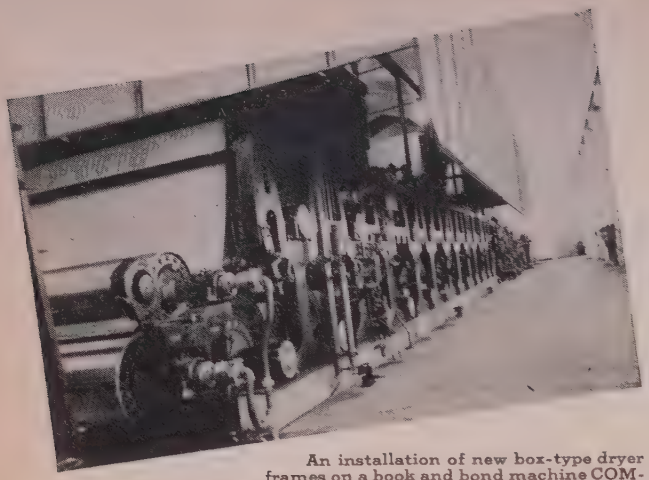
Picture of Gurley Densometer.
Courtesy of W. & L. E. Gurley



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Press section of a completely new machine recently put in operation. COMPLETELY SANDY HILL DESIGNED AND BUILT.

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Possibilities for Development of the Pulp and Papermaking Industry in Alaska

THE purpose of this statement is to provide information to those who may wish to investigate the timber resources, Forest Service timber sale policies, and operating conditions in relation to the establishment of pulp and paper manufacturing enterprises on the Tongass National Forest, southeastern Alaska.

As a result of its studies of the Alaska possibilities for this industry, the Forest Service in 1947 laid out a specific tract of 1,500,000,000 cu. ft. of timber designated as the "Ketchikan Pulptimber Unit" in the vicinity of Ketchikan, and drafted a 50-yr. contract covering timber-cutting rights on this area for the consideration of interested parties. The cutting rights were advertised for sale and on Aug. 2, 1948, a preliminary award of the unit was made to the Ketchikan Pulp & Paper Co., a group connected with the Puget Sound Pulp & Timber Co. of Bellingham, Wash. The successful bidder was required to show reasonable evidence of ability to develop the project and to deposit $100,000 as earnest money which would be forfeited for failure to make the development.

The above preliminary award was later made permanent and assigned to the Ketchikan Pulp Co., a new corporation established under the sponsorship of the Puget Sound Pulp & Timber Co. and the American Viscose Co. Construction of a 300-ton daily capacity plant to make high-grade dissolving pulp was started in June, 1952.

The volume of timber on the Ketchikan Pulptimber Unit is estimated to be sufficient to supply a plant with a daily capacity of 525 tons for a period of 50 yr. The stumpage rates for the timber specified in the award are $0.85 per 100 cu. ft. for material to be used for pulp; $3.00 per M. feet B.M. for spruce logs of no. 1 grade which are used for pulp manufacture, and for spruce logs of any grade going into products other than pulp; $1.50 per M. feet B.M. for cedar; $2.00 per M. feet B.M. for hemlock and other species when used for nonpulping purposes. In addition to the stumpage prices, a deposit of $0.10 per M. feet B.M. for material scaled as sawlogs and $0.15 per 100 cu. ft. for material measured as pulpwood is required for use by the Forest Service on the areas cut over for the improvement of the future stand of timber. Payment for the timber will be made in small installments as cutting proceeds.

The agreement provides that the above rates shall apply for an initial operating period of 10 yr., and gives the Forest Service the right to reappraise and redesignate the rates at the end of that period and at 5-yr. intervals thereafter to bring them in line with then existing timber values, but in no event can the new rates be less than those offered in the original bid. The agreement further provides that in such readjustment of stumpage rates and in the blocking out of areas to be logged in the initial 10-yr. and subsequent 5-yr. operating periods the Forest Service will not take action which will make the price of logs delivered at the pulp mill so high that the purchaser's operations will be placed in a disadvantageous position with respect to similar operations in the Puget Sound region. The purchaser has the right of appeal to the Secretary of Agriculture in this matter, including the right to have a board of three qualified persons review the findings of the Forest Service and submit recommendations to the Secretary.

The Forest Service required in this case, as it will in all cases, that the pulp-manufacturing plant for this timber be established in Alaska.

Other pulp-making enterprises have been or are now being considered by interested groups. These include a proposal for a plant at Sitka, another at Juneau, and still another in the Petersburg-Wrangell area. Figure 1 shows their location and the boundaries of the sustained yield timber areas on the Tongass National Forest from which these plants would draw their timber supplies. The map also shows the location and capacity of water power sites in the vicinity of the mill sites

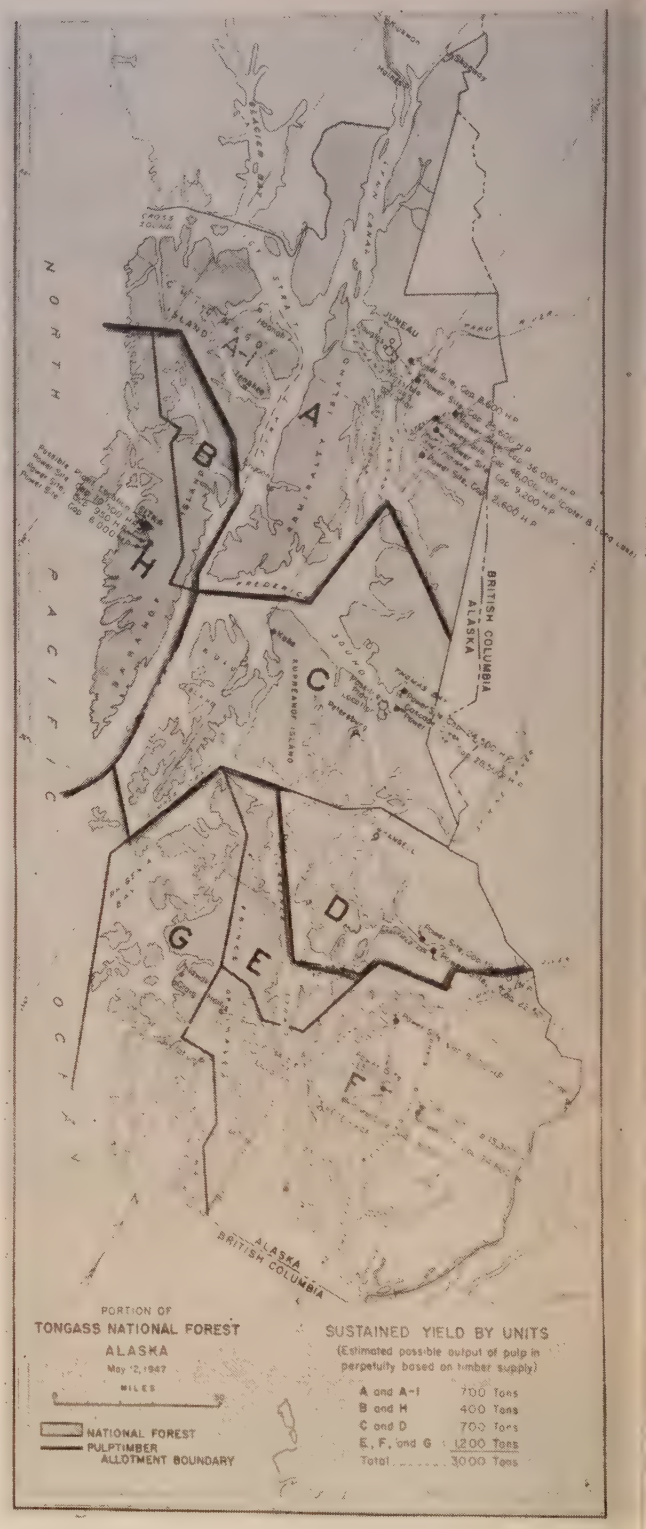

Fig. 1. Sustained yield units in the Tongass National Forest in Alaska

which might be developed to supply power for mill operation

Southeastern Alaska presents an opportunity for the development of permanent pulp and paper-manufacturing enterprises founded on sustained supplies of timber coming from forests that are almost entirely of outstanding pulping species

Prepared by the U. S. Forest Service of the U. S. Department of Agriculture.

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and that are, and will continue to be, under the ownership and management of the Federal Government. The industry will have an almost yearround logging season, cheap log transportation along protected sea channels between the woods and the mill, ocean shipping for inbound mill supplies and outbound mill products, water power for processing, and an equable climate that permits of unhindered mill operation throughout the year. In view of these favorable features, the management plans for the timber resources of the Tongass Forest are so drawn that as rapidly as economic conditions justify the establishment of mills there, these resources will be made available to contribute to a permanent regional development of the industry. The Forest Service will insist that the plans of concerns entering this new field call for economically sized and well-equipped plants, and for operations of a type that will promote continuous employment for skilled woods and mill workers and foster the building up of thoroughly modern industrial communities.

All woods activities will be subject to such Forest Service control as is necessary to keep the production of the forests at a high level.

FEATURES OF THE REGION

Southeastern Alaska consists of a long, narrow strip of mainland and an adjoining archipelago of hundreds of islands, extending southeasterly from the main body of the Territory along the west side of northern British Columbia. The region covers an area about 350 miles long and 120 miles wide. The mainland strip and numerous islands are penetrated and separated by an intricate system of navigable straights, inlets, channels, canals, and bays, which gives the region a total shore line of about 9000 miles and makes a very large proportion of it readily accessible by water. Its total land area is about 34,390 square miles.

This region possesses a high relief and rough topography. The dominant feature is the Coast range, a rugged, snow-capped mountain mass which lies on the mainland and extends the entire length of the region. The islands are made up of ranges of somewhat lower relief. The slopes, both on the islands and on the mainland, are mostly steep and heavily dissected, and the shore lines are largely bold and rocky.

Most of the streams are small since they are on the islands or their catchment basins are confined to the seaward slopes of the mountain range which parallels and adjoins the coast of the mainland.

There are no rail or highway connections with continental United States, except the spur motor road leading out of Haines, at the extreme north end of the region, which joins the Alaska Highway. The nearest transcontinental railroad point is Prince Rupert, B. C., a Pacific Coast terminal of the Canadian National Railroad system, 50 miles from the south boundary of Alaska and 95 miles from Ketchikan. Transportation to and from southeastern Alaska is largely by water, through the sheltered "Inside Passage" which lies back of the island groups that extend from Puget Sound to the north end of southeastern Alaska. Ketchikan and Juneau, the two largest towns, are 660 and 900 nautical miles, respectively, from Seattle. Year-long steamship services is available from Seattle and from Vancouver and Prince Rupert, B. C. The general course from Seattle is northwest rather than north. Ketchikan, the first Alaska port, is almost as far west as it is north of Seattle.

The main sea channels are deep. Ocean-going steamers of almost any size can be used in transporting materials to or products from the existing towns or other sites at which pulp and paper mills might be located. The network of protected sea channels in this region is well suited to the use of motor-driven launches for local transportation. The various centers of population are connected by common-carrier boats of this type, and all industrial enterprises make use of motor-driven work boats. Small tugs with barges and flat scows are used extensively to transport lumber, ore, canned salmon, and other products to and from Puget Sound points. A railroad-car ferry or barge service could easily be operated between the Alaska ports and the railroads at Prince Rupert and on Puget Sound to permit shipment of Alaska pulp and paper to the middle Western States.

The pulp and paper markets of the Orient and Australia are as readily accessible to Alaska as they are to the Pacific Northwest and British Columbia. Those of the Gulf States and other sections of the Atlantic seaboard can be reached through the Panama Canal.

The Pan-American World Airways operates daily scheduled flights between Seattle and southeastern Alaska and a number of local Alaska companies provide daily air service among the towns of the region.

The United States Army operates a commercial telegraph and telephone system between Seattle and the principal towns of Alaska.

In general terms, the Winter climate resembles that of Puget Sound but is somewhat colder. The mean temperatures for the Winter months at the various towns range from 29 to 35°F. Zero temperatures occur infrequently, the official weather records for 30 yr. at Juneau, located in the northern part of the region, showing a total of only 55 days on which the temperature fell to zero or lower. The lowest recorded temperature in Juneau during this same period was 15° below zero.

The precipitation is extremely heavy, the yearly mean for Juneau being 81 in.; for Ketchikan, 156 in. There is no pronounced dry season as in the Pacific Coast States, but the rainfall in autumn, the wettest season, is from two to three times that of an equal period in May, June, and July, the driest months. Snow rarely accumulates to great depths at sea level as much of the Winter precipitation is in the form of rain.

The above statements apply only to low elevations near tidewater, but most of the activities of this region are conducted on or near the coast lines. Winter temperatures decrease rapidly with increase in elevation and progress inland; precipitation is much heavier on the higher mountains and at altitudes above 1000 ft., snow covers the ground for not less than 4 mo. of the year.

There are no climatic factors which prevent or seriously hinder the operation of wood processing establishments throughout the year. The main seaways and most of the small inlets are free of ice throughout the Winter, permitting water transportation at all times. The logging season is usually considered as covering 9 mo., March 1 to November 30, but Winter logging is practicable in many localities so far as weather is concerned. The short Winter days are a handicap, however, as the latitude is high, being about that of Denmark or southern Sweden.

The heavy rainfall of the average Summer gives this region a very low forest-fire risk.

The present population of southeastern Alaska is approximately 30,000, including 6500 native Indians. The principal industry is the sea fishery and its appurtenant service industries. Lumbering and mining are next in importance. The larger towns, together with their estimated present population, are: Juneau, 7500; Ketchikan, 6500; Sitka, 3000; Petersburg, 2000; and Wrangell, 1500. All have community facilities that are at least equal to those of towns of similar size in continental United States. All can be reached by ocean-going steamers throughout the year. Satisfactory locations for pulp and paper mills are available in the vicinity of most of those towns.

Nearly all of southeastern Alaska is in federal ownership and approximately 16,000,000 acres, or 73% of the total area, has been withdrawn from the open public domain and designated as the Tongass National Forest. This forest is administered primarily for the continuous production of

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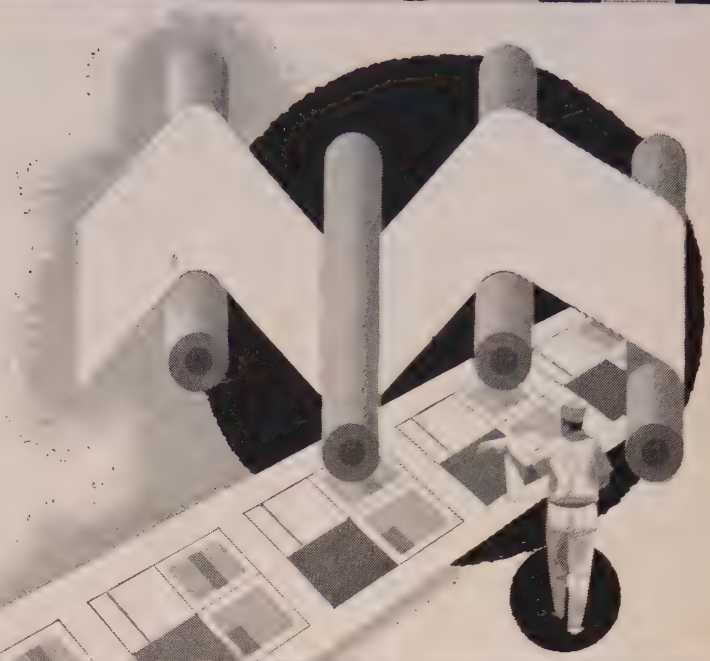

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timber crops and a sustained yearly output of raw material for wood-using industries. Less than 1 % of the total area is potential agricultural land, due to the rough topography and the prevailing thin soils. With the aid of mild temperatures and an abundant rainfall, however, the thin soils are able to support extensive stands of rapidly growing trees at the lower elevations.

Southeastern Alaska carries the extensive "Coast Forest" type of timber which covers the coastal sections of Oregon, Washington, and British Columbia and the southern shores of Alaska as far north and west as Afognak Island. As this Coast Forest pushes northward from Oregon and Washington, it gradually loses some of its species, the timberline decreases in altitude, the trees become smaller, and species of the higher elevations gradually work down toward sea level. In southeastern Alaska it consists predominantly of a mixed stand of western hemlock and Sitka spruce. In many places western red cedar and Alaska cedar occur in the mixture. Any one of these four species may be found occasionally in a pure stand of small extent. Douglas-fir does not extend this far north. The forests are almost tropical in density. In the usual stand, hemlock with some cedar forms the main cover and this is overtopped by trees of the more light-demanding spruce which occur singly or in small groups. Small, bushy saplings of the shade-resistant hemlock and cedar, and blueberry, devil-club, and other shrubs form a dense understory. Down timber, which decays very slowly because of almost continuous saturation from abundant rainfall, is everywhere plentiful.

The forest cover extends from the edge of tidewater to a altitudinal limit of about 2750 to 3000 ft. in the southern part of the region, and 2000 in northern sections. At an elevation of about 1500 ft. the commercial timber usually gives way to stands of dwarfed, limby trees, which are designated "subalpine" and classified as noncommercial. Due to the prevailing steep slopes, the commercial forests form relatively narrow bands along the shore lines of the mainland and islands, rarely extending inland more than 5 miles except along the valleys of the few large streams.

It is estimated that 75% of the commerical timber lies within $2^1/_2$ miles of tidewater.

The forests of commercial value are broken into large blocks by frequent extensive noncommercial areas of "scrub" timber and peat "muskegs." Trees with dead spike tops are a conspicuous feature of the noncommercial scrub and cedar areas. Spike-topped individuals, so decadent as to be classed as unmerchantable, are also fairly prevalent in the overmature hemlock-spruce stands. Few spike tops occur in the mature timber and the piling-sized young timber.

The following represents a conservative estimate of the volume of commercial timber by species on the Tongass National Forest:

Species	*Feet, board measure*	*Per cent*
Western hemlock	58,000,000,000	74
Sitka spruce	15,800,000,000	20
Western red cedar	2,350,000,000	3
Alaska cedar	2,350,000,000	3
	78,500,000,000	100

Approximately 5 million acres of the Tongass National Forest is estimated to carry timber of present or possible future commercial value. At least one third of this area carries timber that is marginal in value at this time, but should become usable in the future. There are also extensive areas of "scrub" timber that is presently unusable, but may some time acquire value.

The average volume per acre of the stands that are classed as commercial under present conditions is between 15,000 and 20,000 board feet, but individual logging units vary widely from this average. Volumes of 40,000 board feet per acre are common over extensive areas. The majority of the merchantable trees are from 2 to 4 ft. in diameter and from 85 to 140 ft. high.

The timber largely consists of fairly even-aged stands and, as in any extensive virgin forest, stands of all ages are present from young reproduction to overmature decadent forests.

FOREST TYPES AND SPECIES

Western Hemlock (*Tsuga heterophylla*)

The western hemlock type is the prevailing commercial timber type of southeastern Alaska. The predominating hemlock has as associates a varying percentage of Sitka spruce, western red cedar, and Alaska cedar. Stands typical of many thousands of acres contain about 75% western hemlock, 20% spruce, and 5% cedar. The type extends from tidewater to elevations between 1500 and 2000 ft. where it gives way to noncommercial stands of mountain hemlock (*Tsuga mortensiana*).

Hemlock is commonly sound when young, but on reaching diameters of 3 to $3^1/_2$ ft., by which time they are largely overmature, trees are affected by disease and rapidly develop spike top and a serious heart rot. Hemlocks occurring on the poorer sites frequently have deeply "fluted" lower trunks, but in most of the commerical forests fluting is not an important factor.

Western hemlock is an excellent wood for a great variety of lumber uses and is superior to eastern hemlock as a pulping wood.

The high value of western hemlock in pulp and paper manufacture has been fully established by the mills of Oregon, Washington, and British Columbia. It is the foundation of the extensive bleached and unbleached sulphite pulp industry which has been developed there in the last 20 years. Much of the bleached output goes into rayon and cellophane which require the highest grades of pulp. As unbleached sulphite and mechanical pulp, western hemlock also provides most of the principal raw material for the newsprint industry of that region. The Forest Products Laboratory, U. S. Forest Service, Madison, Wis., gives the average output of pulp per cord of 100 cu. ft. of solid wood as 2160 lb., moisture-free, by the mechanical process and 1050 lb., moisture-free, by the sulphite process.

Sitka Spruce (*Picea sitchensis*)

The Sitka spruce type is not extensive and its aggregate volume of timber is small in comparison with that of the hemlock type. It occurs in patches which are seldom more than 80 acres in extent and usually much smaller. In practically all cases the spruce stands carry substantial volumes of hemlock.

Sitka spruce occurs from tidewater to an elevation of 1500 ft. In the usual mixed forest the trees of the faster-growing and more light-demanding spruce are larger in diameter than the hemlocks and exceed them in height. The large size and straight, clean, smooth barked trunk make the spruce a very impressive tree. The wood is commonly sound and straight grained, but on exposed sites is much subject to wind-shake and spiral grain. The most common defect of overmature trees is butt-rot.

Stands of the spruce type are supplying most of the present sawmill requirements of southeastern Alaska. The logs are manufactured into all of the usual forms of lumber and also yield a good percentage of high-grade clear lumber that is shipped as a specialty wood to the markets of continental United States. It is an excellent all-purpose pulping wood, comparing favorably in quality and yield with the white spruce of eastern North America.

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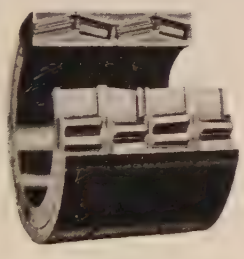

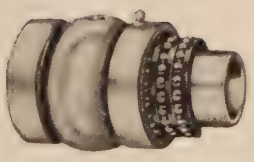

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Western Red Cedar (*Thuja plicata*)

Western red cedar in this region is confined to the southern half of southeastern Alaska and reaches its best development below an elevation of 500 ft. Overmature trees have an average diameter of 5 ft. and a height of 100 to 125 ft. They are limby, heavily tapered, and usually very defective. Trees of pole size are well formed and sound. The best trees are those occurring as scattered individuals or in small groups in mixed stands of hemlock and spruce. The open stands of cedar and other species found so commonly on the swamp soils are uniformly of poor quality and are not considered merchantable, the trees being short, limby, and defective. Western red cedar is used for siding and other high-grade lumber, shingles, and poles. It is also usable for sulphate pulp.

Alaska Cedar (*Chamaecyparis nootkatensis*)

Alaska cedar is found throughout southeastern Alaska from tidewater to the altitudinal limits of tree growth, but is more prevalent and reaches its best development at elevations between 500 and 1200 ft.

The best trees are found in small groups scattered throughout the hemlock and spruce forests. On the wet soils where cedar predominates, the trees are in the main of small pole size and produce a very light volume per acre. The best trees range from 18 to 36 in. in diameter and 75 to 90 ft. high.

Trees over 3 ft. in diameter quite generally carry much heart rot in the first 16-ft. log.

Not much Alaska cedar is now cut as it is not well known in the markets. Its qualities and field of usefulness have not yet been studied carefully, but results obtained in local use indicate a high value for specialized purposes. It has a fine texture, is easy to work, has a bright yellow color, takes a beautiful satin finish, and is extremely durable. It is considered valuable for patternmaking, furniture, toys, turned articles, and cabinet work. A pronounced "cedar" odor makes it valuable for clothes closets and chests. It is used locally for boat-building and telephone poles. Battery separators have been made of it in British Columbia. While costly to log by itself because of its scattered occurrence in the forest specialty uses will doubtless make its utilization profitable.

Scrub

The term "scrub" is used to describe the open stands of dwarfed, defective trees, with dense undergrowth of blueberry and other shrubs which occupy the soils more poorly drained than those of the forest types previously described but better drained than those of the muskeg type. Many of the trees are spike-topped and highly defective, have a rapid taper and long, ragged crowns, and in general present a scrubby and unhealthy appearance. All of the coniferous tree species of the region may be represented, but the cedars usually predominate. Stands of scrub generally have many interspersed areas of muskeg, one type merging gradually into the other. Clumps of fairly good trees are scattered throughout the stands and may sometime be logged as cordwood.

Muskeg

The muskeg type consists of a vegetable cover of moss, grass, herbs, and a few isolated stunted trees, including lodgepole pine (*Pinus contorta*), growing on peat accumulations often 5 ft. or more in thickness. It covers extensive areas in this region.

SILVICULTURAL FEATURES

Sitka spruce is a rapidly growing tree and reaches its best development when growing in mixture with other species, which it always overtops. Western hemlock, its principal associate, has fair growth properties and is shade enduring, a quality enabling it and the more light-demanding spruce to form dense mixed stands. Studies made to date indicate that the two are an excellent combination for the production of pulpwood, and that the aim of the forester should be to reproduce the mixture, but with an increase in the proportion of spruce.

The original forest cover in this region was doubtless almost pure spruce, but such stands gradually tend to give way to hemlock, the climax type. Areas recently uncovered by receding glaciers show spruce as the predominant species, but with hemlock beginning to invade the stand. Over most of the region the first growth is older and hemlock now forms the major portion of the mixture. It is thought that under forest management this natural tendency toward pure hemlock can be checked and the proportion of spruce in the regrowth on the cutover areas materially increased through the use of methods that favor the latter.

Clear cutting with reserved seed trees is the silvicultural method now practiced in the sawtimber operations which characterize logging here, and ordinarily the areas so logged show ample reproduction and an increase in the proportion of spruce in the stand to 50% or more. Clear cutting also obviates much windfall, a serious danger with these shallow-rooted species. Due to this danger, seed trees on the clear-cut areas are not left as scattered individuals, but in fairly large groups. This method has the added advantage of protecting the seed trees from damage by the heavy machine logging which must be practiced here. If further study should show that favorable results can be obtained by individual tree or group selection methods of cutting on certain classes of sites, such methods may be adopted for those sites.

It does not appear at this time that the disposal of logging slash on pulpwood cutting areas need extend further than lopping and scattering of tops so that they lie close to the ground. Even these simple measures may be found unnecessary on the average logging area. The fire hazard is usually so low here because of the wet climate that expensive slash disposal work is ordinarily not required from that standpoint.

Preliminary studies of yield in the forests of southeastern Alaska indicate that the rotation should be around 80 yr. and that the crop produced in this period will have a volume per acre well above the average volume now found in the virgin commercial forests.

With an estimated commercial stand of virgin timber on the Tongass Forest of 78.5 billion board feet and allowing an average of 78 yr. as the rotation period during which this virgin timber may be entirely removed under sustained-yield forest management, approximately 1 billion board feet or 2,000,000 cords of wood can be taken from the forest each year in this period. If devoted to pulp manufacture, this output would be sufficient to produce approximately 1,000,000 tons of sulphite pulp a year.

WATER POWER RESOURCES

The potential water power resources of southeastern Alaska include 800,000 hp. of firm power which has been covered by reconnaissance or more detailed surveys to date. Further power explorations are almost certain to disclose additional important sites. Outstanding characteristics of the power sites are: high-head developments, short conduits, small drainage basins with heavy runoff, good water storage facilities, accessibility of the projects to navigable waters, and the opportunity to locate industrial plants either directly at the powerhouse sites or within short power-transmission distances.

There are no very large capacity power sites. The largest single site so far investigated has a year-long capacity of 32,000 hp. However, in many places, power from several sites can be economically concentrated at one manufacturing point. For example, 50,000 hp. can be developed in one powerhouse from two sites adjacent to the head of Speel River

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Arm of Port Snettisham near Juneau, and the development of two other sites in the same locality would increase the concentration at this point to a total of 75,000 hp. Three sites adjacent to the head of Bradford Canal near the town of Wrangell have a combined capacity of 54,000 hp. A concentration of 60,000 hp. can be effected at Ketchikan.

The typical water power site has a high "hanging" lake located a short distance inland from tidewater. This can be made to provide storage by use of a dam across the lake outlet, a tunnel to tap the lake below its present water surface, or a combination of these two methods. Many of the lakes have elevations exceeding 500 ft. Conduits to connect the lakes with powerhouses at tidewater will range from $^1/_2$ to 3 miles in length, and in most cases will be less than $1^1/_2$ miles long.

The runoff per square mile of drainage basin is high because of heavy precipitation in this region. The normal period of low stream flow occurs in Winter when the precipitation on the high watersheds is largely in the form of snow, and extensive water storage for Winter use is necessary to obtain the regulated flow throughout the year which is essential for a pulp and paper plant. All of the water power sites considered suitable for pulp and paper projects have lake storage and the possibility for such regulation of flow.

The Federal Water Power Act provides for the development and use of power sites under a form of license covering a period not in excess of 50 yr. and calling for an annual rental fee per developed horsepower. Licenses are renewable under certain conditions at the end of the period for which granted.

LIMESTONE AND COAL

High calcium limestone deposits are available at tidewater points in southeastern Alaska. Coal can be obtained most readily from mines on Vancouver Island or in western Washington. Oil is received by tanker from the California refineries.

LOGGING METHODS AND COSTS

The methods of logging required in Alaska are similar to those used in western Washington and Oregon and the necessary machinery and supplies are manufactured in those states. Machine logging with donkey engines and wire rope has proved to be the most practical means of moving logs from the stump on the usual logging area in this region because of the rough topography and wet ground. Ground-skidding and high-lead systems are now used, but one of the overhead systems will probably prove most economical for extensive pulpwood operations.

A large percentage of the timber can be logged directly into tidewater by the use of two and three donkey engines working tandem. Motor truck roads will be required to transport the timber from the donkey yarding engines to tidewater in the longer valleys. Log driving is not practicable in the short turbulent streams.

Timber is commonly handled in full tree lengths or as very long logs from the stump to the mill. After being placed in tidewater, it can usually be towed in the form of flat rafts through the protected waterways. Davis-type rafts are required for Winter towing in the wider, more exposed channels. The cost of towing sawlogs is about 2 cents per 1000 board feet per mile. The cost per 100 cu. ft. for pulpwood logs should not exceed this figure and may be somewhat less. The timber supply for a plant can thus be drawn cheaply from a very extensive region.

Floating logging camps which can be towed from one cutting area to another are in general use. Similarly, donkey engines and all other logging equipment are moved on scows and floats. The average cost of sawlogs, exclusive of stumpage, delivered at the sawmills just prior to the war was about $10 per 1000 board feet. It is estimated that on the basis of labor and equipment costs of 1951 the *total* cost of unpeeled pulpwood logs delivered at local mills would not have exceeded $13.50 per 100 cu. ft. (equivalent to $27 per 1000 board feet).

FOREST ADMINISTRATION

Practically all of the timber manufactured in southeastern Alaska is purchased from the national forest. The amount taken yearly from the forest under commercial sales is now about 60,000,000 board feet. Most of this goes into lumber, but about 20% is used in the round for fish-trap and wharf-piling. Ketchikan has a modern electric-driven sawmill of 100,000 board feet capacity per 8-hr. day. Sitka has an efficient mill of 35,000 board feet capacity, and smaller mills are found in other communities. A 75,000-ft. mill is nearing completion at Metlakatla.

Alaska has no operating pulp or paper plants at this time. The nearest mill is the dissolving pulp mill of the Celanese Corp. at Prince Rupert, B. C. The newsprint plant of the Pacific Mills (Ltd.) is located at Ocean Falls, B. C., 296 miles south of Ketchikan.

The national forests of Alaska are administered by the Forest Service, U. S. Department of Agriculture, with a staff resident in Alaska. Only matters involving questions of important general policy need be referred to Washington. The chief administrative officer is the Regional Forester with headquarters at Juneau. The Tongass National Forest is divided into three divisions with headquarters at Juneau, Ketchikan, and Petersburg, and with a Division Supervisor in charge of each. Suboffices are located at Sitka and Craig. The Regional Forester has assistants for handling specialized and technical lines of work.

A fleet of seagoing launches is maintained to provide field transportation for Forest Service personnel.

The following is a condensed statement of the main objectives and timber-use policies which have been established by the Forest Service for the management of the timber resources of the Tongass National Forest:

Objectives: The development and maintenance of a permanent pulp and paper manufacturing industry based on a sustained yield of the timber resources.

The furnishing of a permanent and convenient supply of timber for local consumption with such an additional supply to the local sawmills for processing for the general lumber markets as may be needed to justify efficient milling facilities and provide year-long operations.

Timber-Use Policies: The productive forest land will be divided into pulp-timber allotments, local-use allotments, and general-use areas.

Pulp-timber allotments will be laid out to supply the timber needed in the establishment of pulp and paper mills of economical operating size in each section of the region. Sustained-yield timber management will be practiced on each allotment or group of allotments.

Local-use allotments will be laid out on the basis of the suitability of the timber for the common local uses and its accessibility to points of consumption.

General-use areas will include all bodies of timber not specifically placed in the two foregoing classifications They will be available for sale for any product to be manufactured in the Territory for the general or local market.

So far as the Forest Service is able to control the location of pulp mills in southeastern Alaska, they will be so distributed that an adequate timber supply under the management plan will always be available within a reasonable log-towing distance of each plant.

A condition of every large sale will be that the wood must be given at least primary manufacture in Alaska. Other things being equal, preference will be given to enterprises that contemplate the most complete manufacture in Alaska.

The establishment of minor wood-using industries, especially those using western red cedar and Alaska cedar, will be encouraged.

Farrel calender rolls are the product of a century of roll-making experience, highly specialized plant facilities, skilled metallurgical and mechanical engineering, and exacting methods of quality control. When you *specify Farrel rolls* you will get:

1. The correct metal formula for the service in which they will be used.
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Farrel rolls are made in *the world's largest specialty roll shop,* in any size from 5″ to 72″ in diameter and up to 312″ face length. For further details, send for a copy of Bulletin No. 116. No cost or obligation.

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GRID is an all cast unit with finned heating sections and headers tested to withstand steam pressures up to 250#P.S.I.

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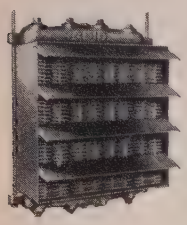
GRID construction has all similar metals in contact with steam, thus preventing electrolytic corrosion which eventually occurs in copper type unit heaters where steam passes from iron pipes into copper cores. (Write for booklet CORROSION IN UNIT HEATERS).



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No sales except for purely local use will be made to aliens or alien corporations.

Small sales will be encouraged so far as is consistent with the investment required.

Preservation of Scenic and Recreational Values—Protection of Salmon Streams: Scenic and recreational values rank high in much of the national forest area; predominate over considerable areas, and must be adequately safeguarded.

All timber management plans will be carefully coordinated with plans for the preservation of the scenic and recreational resources of the forest and the protection of salmon spawning streams.

Timber sales will not be made in locations where the scenic and recreational values are highly predominant. Established recreation areas will be held intact. Along heavily traveled narrow sea channels and in other locations where justified, sales will not be granted or they will be permitted only under some form of selective logging which will prevent unsightly scars or openings in the timber cover, or exposure of the remaining stand to probable windfall. Within the boundaries of timber sale areas, blocks of timber will be left standing along the shorelines of important recreation lakes and fishing streams and along main roads. Logging will be so conducted that salmon streams will not be damaged through mechanical injury, logging debris, or siltation from soil erosion.

General Conditions Applying to National Forest Timber Sales

Timber-cutting rights only are offered for sale on the National Forests, the land being retained by the United States for the production of successive forest crops. The maximum amount of timber that will be placed under contract to one firm and the period of time allowed for its cutting will be determined by the Forest Service in consideration of the amount of the purchaser's logging and manufacturing investment, practical operating methods, and available markets.

The timber unit to be offered for sale is appraised by a forest officer on the basis of its value for manufacture into the timber products which offer the best markets. The unit is then advertised for sale by oral or sealed bids for a period of not less than 30 days, the advertisement naming the appraised stumpage rates as the minimum that will be considered. It is awarded to the highest bidder who can make a satisfactory showing of ability, financial and otherwise, to carry the proposed operations through to completion.

The timber is paid for in advance installments as cutting proceeds and on the basis of a scale or measurement of the cut material made by a forest officer. The amount of each installment is approximately equal to the value of the stumpage expected to be cut by the purchaser in a period of 2 to 3 mo.

The bid stumpage rates in every long-term sale apply for a limited period only and are subject to readjustment at stated intervals thereafter throughout the life of the contract to keep them in line with possible changes in the value of stumpage.

Some other contract provisions, notably the utilization requirements, are subject to modification on the dates when the stumpage rates may be readjusted.

National forest timber cannot be held for speculation or other purposes. Contracts specify a definite date by which logging must begin, and provide that certain minimum amounts be cut in given periods. Performance bonds are required of purchasers. Contracts can be assigned with the approval of the Forest Service.

Timber is sold on the basis of the unit customarily used in the industry concerned. Thus, timber to be used for sawlogs or veneer blocks is sold by the board foot using the Scribner Decimal C log rule; piling and poles by the linear foot, etc. The pulp-timber unit widely used in the industry is the cord, but as pulpwood in Alaska will ordinarily come to the mills in long logs and not be reduced to cord wood form, the cubic foot has been selected as a simple, suitable unit, easily applied. The price unit for pulp-timber is 100 cu. ft. of solid wood (excluding bark and rot), an amount approximately equal to a cord. One hundred cubic feet is equivalent to between 500 and 600 board feet in average-sized logs in Alaska.

Alaska contracts usually provide for "clear cutting" the merchantable timber on the sale area with the exception that not to exceed 10% of the volume, in strategically placed patches selected by the Forest Service, will be reserved for natural reseeding purposes. These groups of seed trees must be protected against injury from logging operations. Some form of selective cutting may later be required in situations where found to be feasible. If natural reseeding fails on any particular area, the Forest Service would expect to reforest it by planting.

The disposal of logging debris to the extent of lopping the tops and scattering the brush so that it lies close to the ground and away from seed trees and clumps of reproduction may be required of the purchaser where such action appears desirable as a fire protection measure. Satisfactory precautions must be taken against the escape of fire from logging operations.

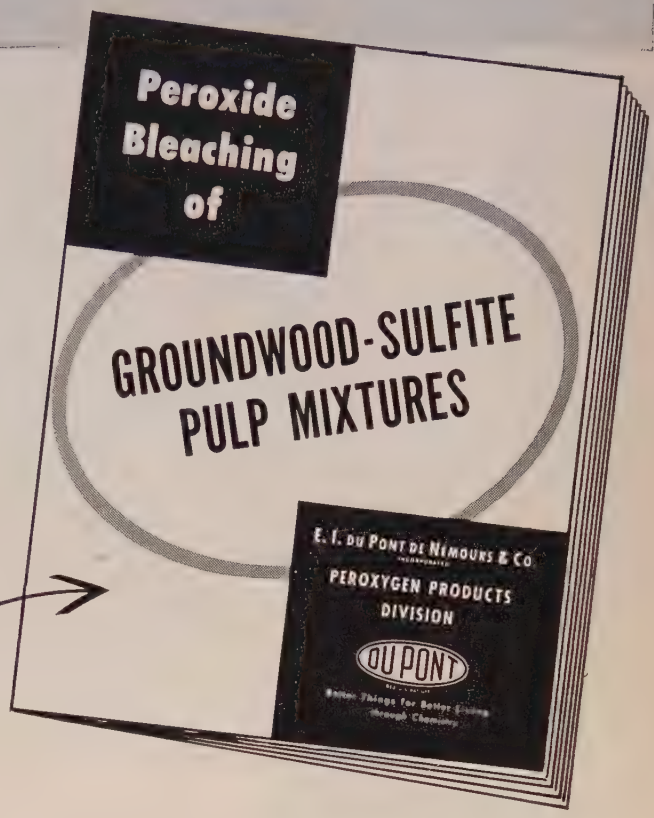
All logging activities must be so conducted that the forest is left in good condition for regrowth.

In addition to payments for stumpage, the purchaser will make deposit of specified sums with the government to help defray the cost to the Forest Service of work to be done on the cutover areas to improve the future timber stand.

Logging operations must not interfere with salmon spawning streams.

How to produce premium grades of printing paper at low cost

Upgrade both groundwood and sulfite pulps in one peroxide bleaching operation



Peroxide bleaching of groundwood-sulfite mixtures enables mills to process the entire paper furnish in one bleaching operation . . . reduces capital investment for bleaching equipment and lowers operating costs. Bleached pulps produced with this process feature superior brightness, cleanliness, absorbency and paper machine formation characteristics . . . are ideally suited to the economical production of premium grade printing papers.

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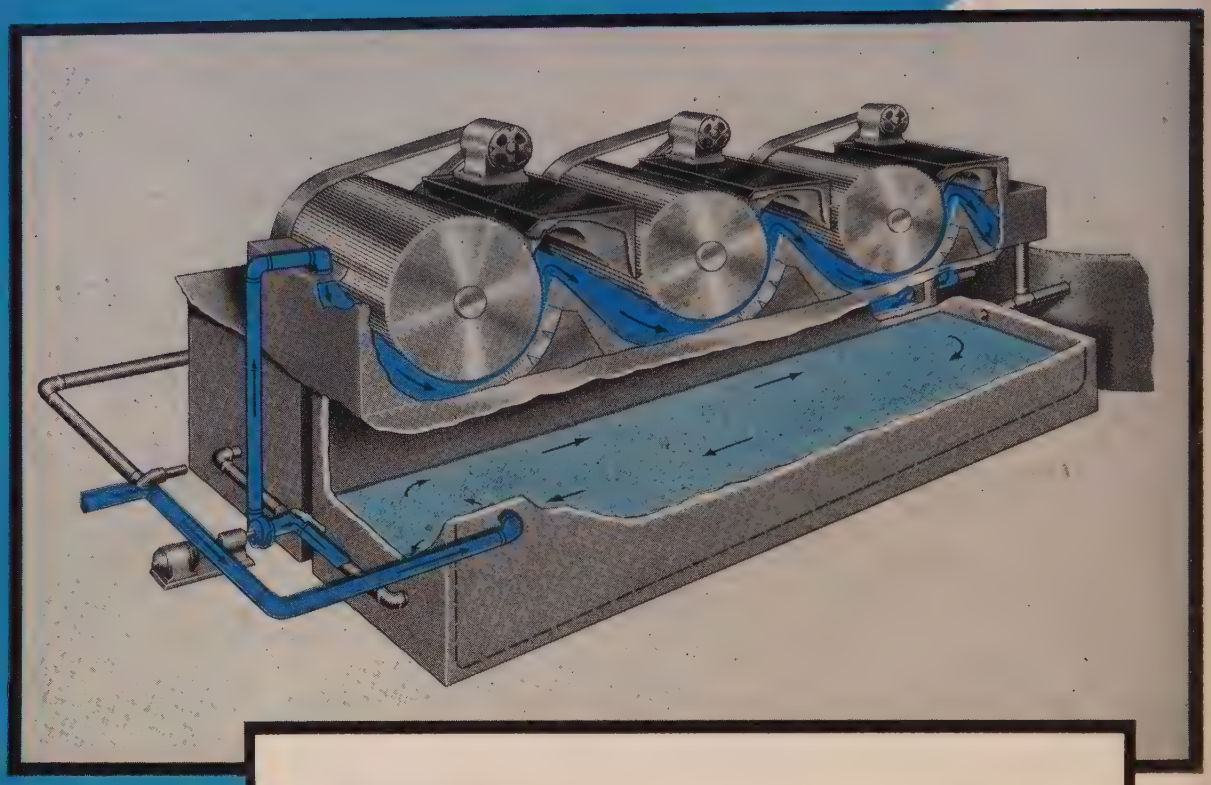
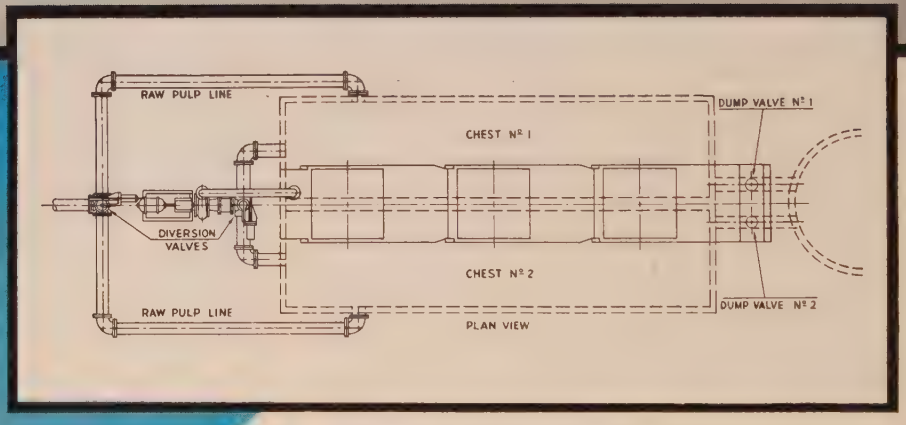
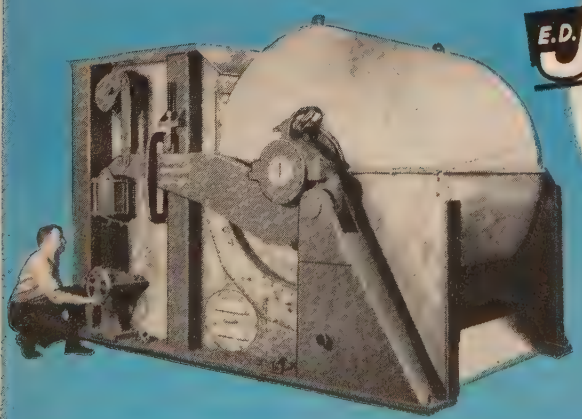




LEGEND

Raw stock is admitted to Chest #1. Diversion valves, controlled by liquid level elements, automatically shut raw stock line, circulate stock from Chest #1 through Multi-Beater and into Chest #2; then again through Multi-Beater back into Chest #1, repeating cycle as required.

On last pass, refined batch is automatically diverted to beaten stock chest at the same time the next batch of raw stock is being admitted to whichever of the two chests is empty, and new cycle begins without pause.



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This easy-to-install "packaged" Beating Unit is the heart of the *Continu-flo* system. • Its pre-assembled construction means lower installation cost—no assembly in the mill. • Its proved mechanical design embodies the best features of the famous Jones-Bertrams Patent Beaters — produces better quality stock at lowest cost per unit of refining capacity. • And its integral motor support saves floor space. Available for either tub or multi-beater installation.

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Double-chest Batching System

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Batch-refining quality with the tonnage and economy of continuous operation

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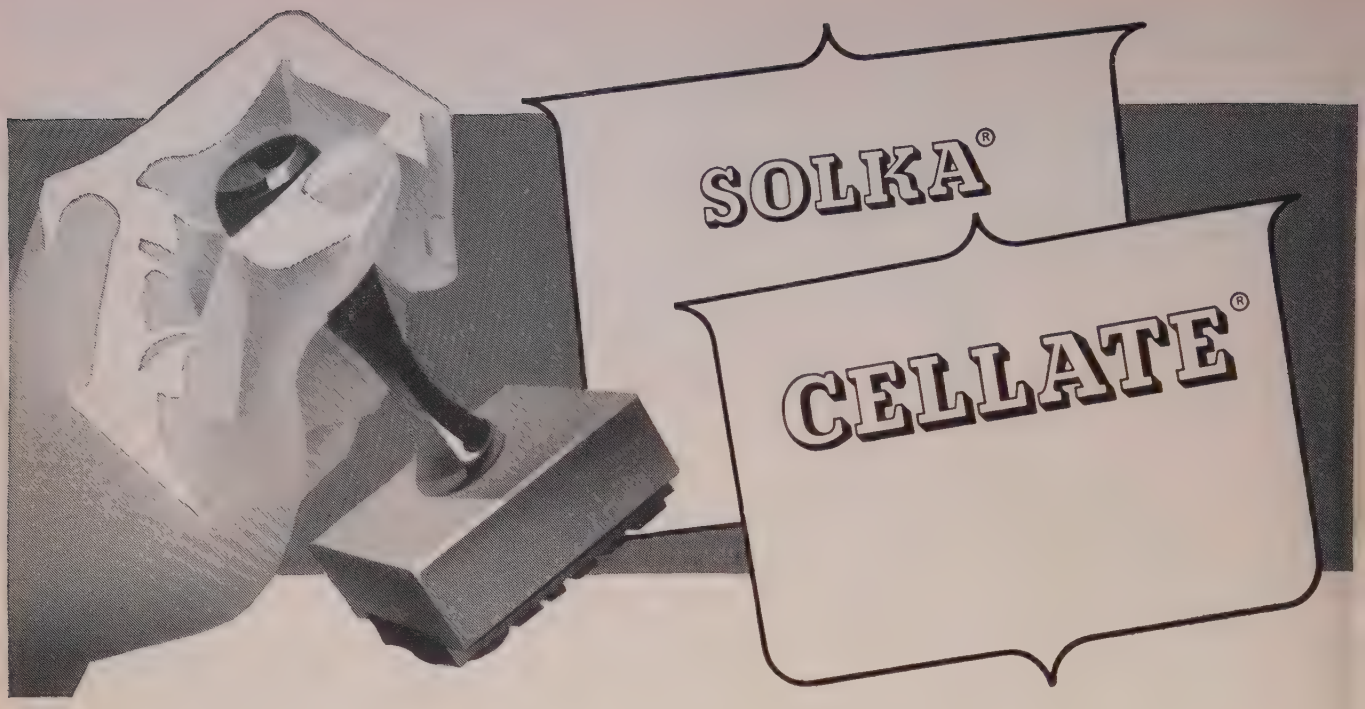

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Original arrangement with Tra fer Press and combination 3-R Main Presses (Cloverleaf).

Modified arrangement with Tra fer Press and conventional M Presses.

Modified arrangement with co bination Transfer and Main Pr (Multipress) and conventio Second Press. Minimum sp required.

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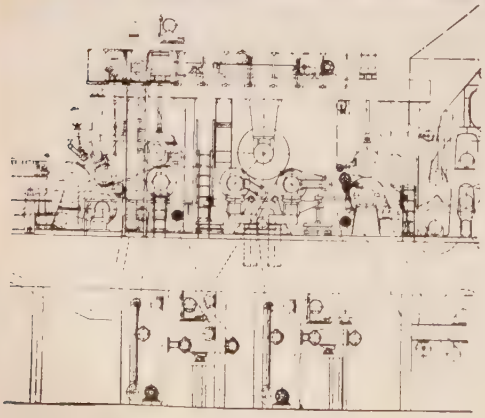
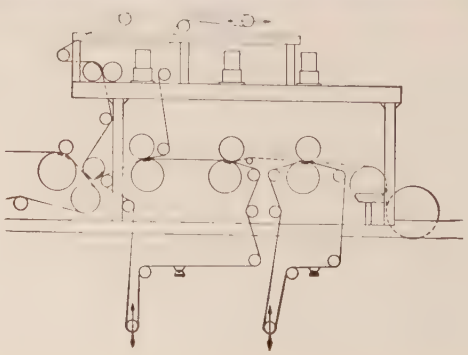
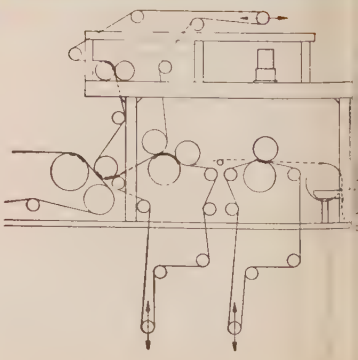
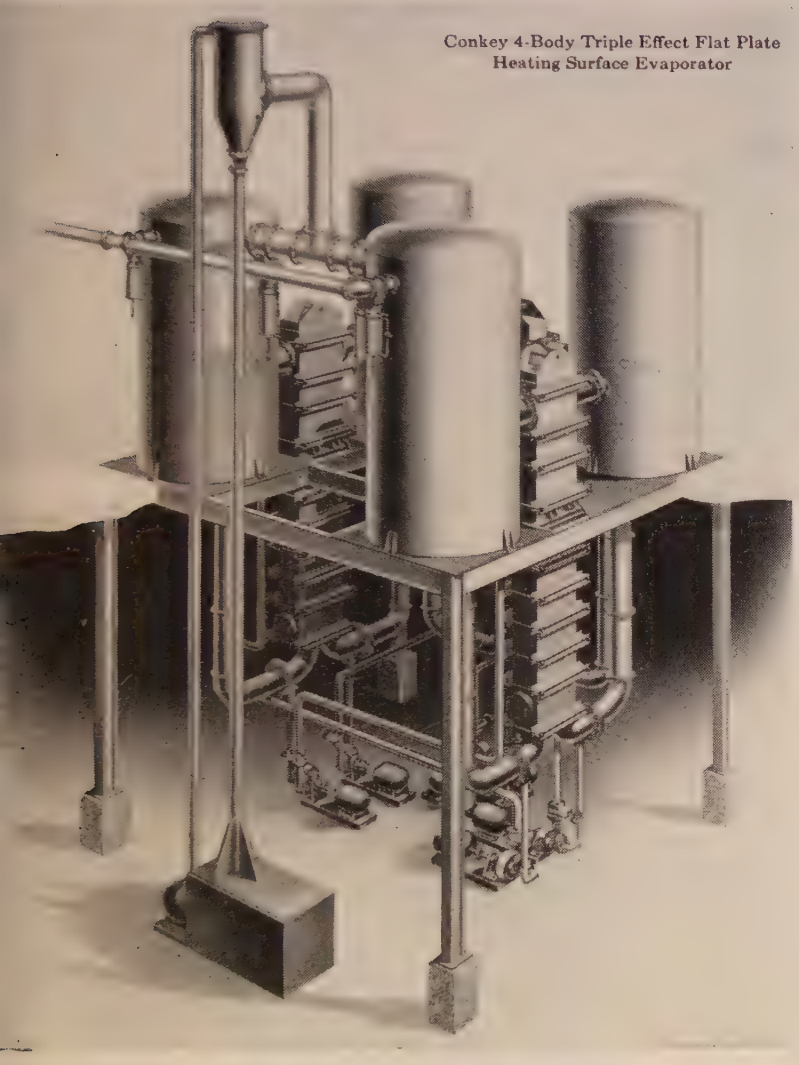






NO shut down time! stream pollution! scale removal costs!



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Conkey Flat Plate Heating Surface Evaporator *with Rosenblad Switching System**

The proven system for avoiding stream pollution by sulphite pulp mill waste liquors that proved so outstandingly successful in commercial installations all over Scandinavia, is now adapted for use in this country by General American. *In every instance* where a Rosenblad Switching System has been installed, shut down time *and* scale removal *costs have been practically eliminated!* The Rosenblad System utilizes the condensate wash as a descaling operation carried on during full capacity operation of the evaporator. Surfaces subjected to boiling liquor are periodically switched with those in contact with vapor and condensate to clean heating surfaces during normal continuous operation. *Every part* of the equipment is switched, consequently scale is washed away from pipe lines, valves and vessels . . . in addition to heating surfaces.

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Take Pulp and Paper Mills for example. Few industries use more steam or have a larger stake in the economy and reliability of their steam generating equipment. The list below is just a sample of the nationally-known leaders in the pulp and paper field that have Vertical-Unit Boilers in service at one or more mills.

Only larger companies are listed because here, as in any industry, the buying decisions of big companies are especially significant. Such companies have the breadth of experience, the diversified operating conditions and the organization necessary to explore and evaluate the merits of the equipment they need.

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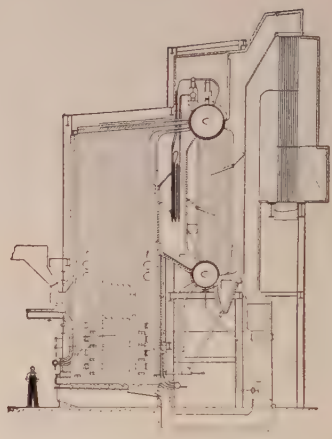
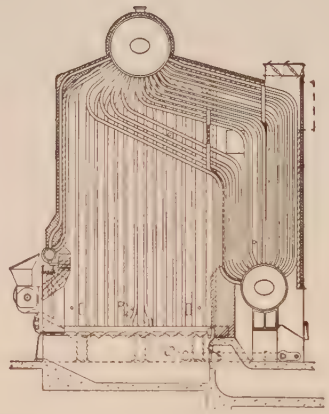
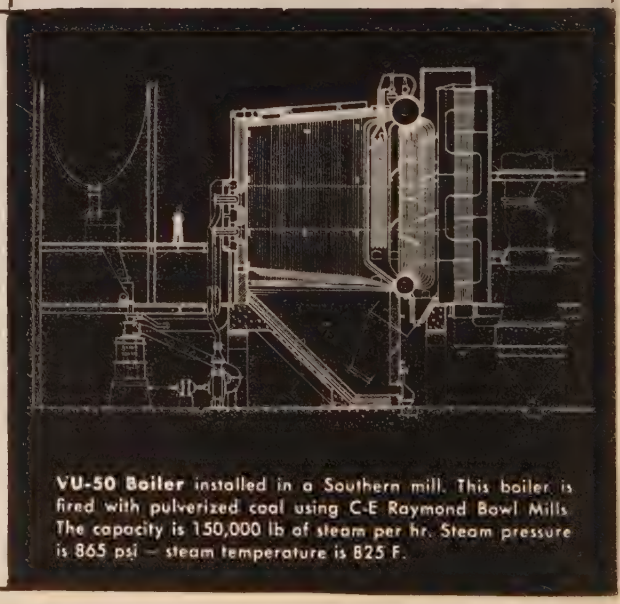
Macon Kraft Corporation
Marathon Paper Mills of Canada, Ltd.
National Container Corporation
St. Regis Paper Company
Scott Paper Company
S. D. Warren Company
Weyerhauser Timber Company



VU-50 Boiler — This unit, one of two duplicates, is installed at a West Coast mill. It is designed to burn hogged wood on a high-set spreader stoker. Capacity — 150,000 lb of steam per hr; operating pressure — 570 psi; steam temperature 675 F.



VU-10 Boiler, one of two in a Midwestern plant. Fired by coal, using a C-E Spreader Stoker. Capacity — 18,000 lb of steam per hr at 200 psi; no superheat. VU-10 capacities range from 10,000 to 60,000 lb of steam per hr.



VU-50 Boiler installed in a Southern mill. This boiler is fired with pulverized coal using C-E Raymond Bowl Mills. The capacity is 150,000 lb of steam per hr. Steam pressure is 865 psi — steam temperature is 825 F.

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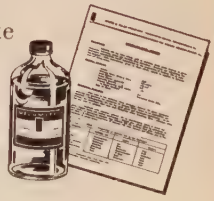

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The Characteristics of Pulp Fibers from Agricultural Residues

ELBERT C. LATHROP

PERHAPS the most critical problem presented by the rapidly increasing world demand for cellulose pulps is the supply of softwoods. The long-fibered pulps produced from them are essential for the manufacture of wrapping and other high tear-resistance papers. With few exceptions, stands of the species are found only in the Northern Hemisphere and without considering Siberia, the extent of softwood forests is known.

In the United States, in Australia, and to a limited extent in Europe, there is an increasing production of cellulose pulps from certain hardwood species. Blends of softwood and hardwood pulps to produce a variety of paper and board products are becoming standard practice in many mills. A study of tropical woods as a source of cellulose pulp is of extreme importance.

This paper is designed to show how a partial, practical, and economic solution to the problem can be given on the basis of the chemical and physical characteristics of pulp fibers from agricultural residues. Examples of applied research will be described which point the way to success in this field.

USE OF ANNUAL PLANTS FOR PAPERMAKING

Annual plants have been used in limited quantities to produce cellulose pulps for more than a century. Examples are esparto, used mainly for fine printing papers by the English, the tow of seed flax straw to produce cigarette and fine book papers in the United States and Canada, and wheat straw to produce corrugating board and fine bleached pulp, used mainly as blends with wood pulps to make fine papers. *Arundo donax* is used in Italy to make alpha-cellulose, and sugarcane bagasse is used as a blend with wood pulps for various grades of paper in Peru, in Argentina, and in the Philippine Islands, with new mills being build for this purpose in India and in Brazil. Bamboo is used in India and Argentina. Old rope is pulped for use in making the strongest bag papers.

In discussing the characteristics of pulp fibers from agricultural residues, the bearing of their unique physical and chemical properties on practical papermaking is of main importance.

TYPES OF FIBERS AVAILABLE

Based roughly on chemical composition, fiber dimensions, density of raw material, and ease of pulping, those plants that have received most industrial attention as raw materials for pulpmaking fall into the five groups shown in Table I.

Table I. Grouping of Agricultural Fibers

Group	*Fiber*	*Plants chiefly used as pulp sources*
1	Straws and esparto	Wheat, rye, rice, esparto
2	Canes and reeds	Sugarcane bagasse, *Arundo donax*, cornstalk, *Phragmites communis* Trin.
3	Woody stalks with bast fibers	Flax, hemp, cotton, soybean
4	Leaf fibers	Abaca (manila), sisal, henequen, pineapple, caroa
5	Bamboos	Various varieties

CHEMICAL COMPOSITION AND PROPERTIES

Table II shows the approximate variations in the principal chemical components of these plants or plant fibers. From these data it will be noted that the annual plants contain much more ash than do the pulpwoods. The cereal straws, particularly rice straw, have high ash contents.

In lignin content, the bast and leaf fibers are quite low; it is high in all pulpwoods, bamboos, and woody stems of plants producing bast fibers. The cereal straws and esparto contain less lignin than the stalks and reeds, but the plants of both groups 1 and 2 are lower in lignin than the pulpwoods, bamboos, and woody plant stems. These differences are reflected in the amounts of chemicals required for producing fine bleached pulps from the members of the various groups.

The pentosan content of the bast fibers is very low while the straws, esparto, stalks, and reeds analyze highest in this component. However, the deciduous pulpwoods and the leaf fibers approach the straws in pentosan content and decreasing amounts are found in the woody stems, bamboos, and coniferous pulpwoods.

Cross and Bevan and alpha-cellulose contents are highest in the bast and leaf fibers, decreasing slightly in the order, bamboos, coniferous woods, stalks, and reeds. The deciduous woods contain slightly more cellulose constituents than straws and esparto, and woody stems contain the least. Pith-free bagasse fiber compares favorably in cellulose contents with coniferous woods. The cellulose content of pith (parenchyma) is lower than that of the fiber. Bamboos analyze higher in Cross and Bevan and alpha-cellulose than the coniferous pulpwoods.

Data regarding the holocellulose content of all the material under discussion do not seem to be available. However, with the probable exception of bast and leaf fibers, the holocellulose content for the other fibers, on an ash-free basis, would appear to be at fairly uniform level near 70%. This suggests that differences in the fibrous carbohydrate materials in many plants are qualitative; that is, they differ in kind rather than in amount.

PHYSICAL PROPERTIES AND DIMENSIONS

Variations in fiber dimensions are listed in Table III. As is well known, the bast and leaf fibers are much longer than those of any other groups. Coniferous woods and bamboos possess fibers of substantially the same length. Such fibers produce high tear resistance in papers and, in fact, possess near the maximum length practical for good papermaking. The deciduous woods, straws, esparto, stalks, and reeds have somewhat shorter fibers. They vary about the same in length among the various groups as do the individual fibers within the groups. The tearing strength of papers made from them alone is low and such papers are not so suited for wrapping and bags, when papers made of longer fibers are available. Fibers of the woody stems of plants in group 3 are very short and entirely unsuited for tear-resistant papers.

It is now generally recognized that the ratio of fiber length to fiber diameter is one of the most important criteria for evaluating papermaking fibers. With the exception of the fibers of the woody stems, this ratio is higher for all agricultural fibers than those of the pulpwoods. The values for bast and leaf fibers are exceptional. The ratios for rice straw and bamboos are about twice those of coniferous woods, which are also exceeded by straws, esparto, and some of the reeds. Sugarcane bagasse fibers compare very favorably with the conifers in respect to this ratio. The fibers of the deciduous woods as compared with those of groups 1 and 2 show less than one half to one third of their ratio, while the ratio in the case of the woody stems is exceedingly poor.

PULPING CHARACTERISTICS

Table III classifies the various groups of fibers in relation to density and to ease of pulping. Fibers in group 1 are easiest to pulp because of the very open plant structure and their relatively low lignin content. The chemical reactions involved

ELBERT C. LATHROP, Chemical Engineer, Head of Agricultural Residues Division, Northern Regional Research Laboratory, one of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture, Peoria, Ill.

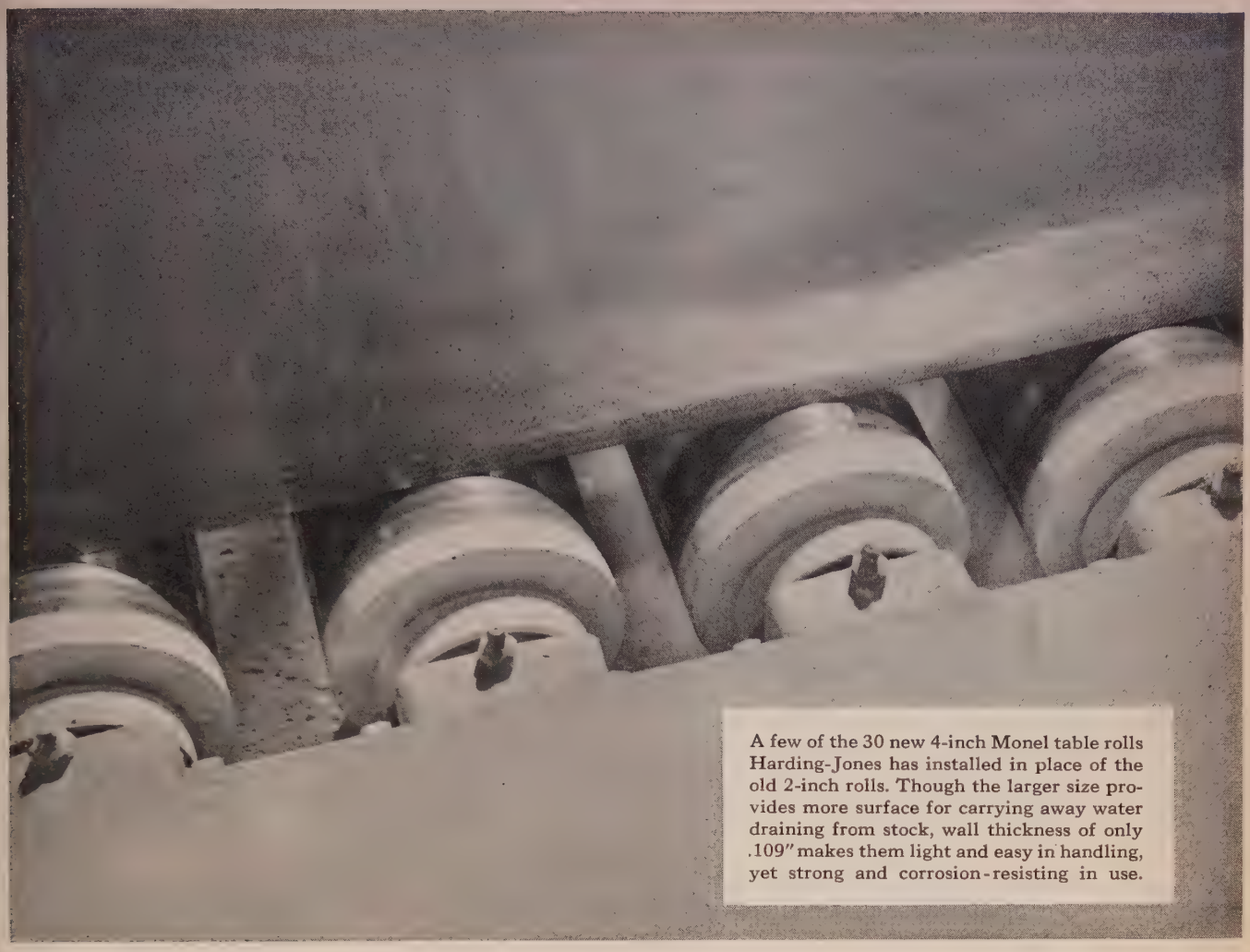

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Table II. Agricultural Fibers and Pulpwoods; Variations in Principal Chemical Components

Group	Fiber	Ash, %	Lignin, %	Pentosans, %	Cross and Bevan cellulose, %	Alpha-cellulose, %
1	Straws and esparto	6–8	17–19	27–32	50–54	33–38
	Rice straw	14–20	12–14	23–25	46–49	28–36
2	Stalks and reeds	3–6	18–22	28–32	52–58	33–43
	Sugarcane fibers	2	19–21	30–32	59–62	40–43
3	Woody stalks with bast fibers					
	A. Woody stems	2–3	23–27	15–22	40–48	31–33
	B. Bast fibers	1–2	1–6	2–6	70–80	60+
4	Leaf fibers	0.6–1.2	7–10	17–24	70–80	53–64
5	Bamboos	1–2	24–29	16–18	60–63	50+
6	Coniferous woods	<1	26–34	7–14	53–62	40–45
7	Deciduous woods	<1	23–30	19–26	54–61	38–49

are undoubtedly surface reactions. The open structure and thin walls of the straws and esparto present a large initial surface for chemical attack and minimize the problem of diffusion encountered with all of the pulpwoods. The pulping reaction is therefore rapid and the chemicals required for pulp production are lower than with other fibers.

Next in order come the canes and reeds. These plants require somewhat more chemicals and somewhat more vigorous pulping conditions. This is due to the fact that the rind fibers of these plants are generally denser and the fiber walls thicker than those encountered in group 1. In some cases, especially with sugarcane bagasse, the lignin content may be higher. For example, in Florida sugarcane, which is harvested 9 to 11 months after planting, the lignin content was 18 to 19% while Hawaiian cane cut after 22 months contained from 22 to 23% lignin. Naturally, more chemicals are required to pulp this particular Hawaiian bagasse.

Bast and leaf fibers frequently present a problem in pulping because of associated impurities, but such fibers alone, being very low in lignin and high in cellulose, pulp with greater ease and with less chemical requirements than pulpwoods, bamboos, and the fibers of woody stems.

These data show that industry is not limited to the conifers to obtain fibers of optimum length for making tear-resistant papers. Bamboos provide fibers similar in length while the bast and particularly the leaf fibers provide fibers of greater length. Indeed, it is well known that leaf fiber pulps (abaca) are now used as blends with wood fibers to make the strongest of our bag and wrapping papers. These fibers, as well as bast fibers, are used in the manufacture of currency, bank note, and high tear and scuff-resistant papers. In many cases the length of the fibers of straws, esparto, canes, and reeds equals or exceeds that of the deciduous woods, and from the viewpoint of producing strong papers their pulps often exceed pulps made from hardwood species.

PRACTICAL PROBLEMS IN PULPING AGRICULTURAL FIBERS

The overwhelming preference of the pulp and paper industry for wood fibers is partly due to inertia. This has been generated in the minds of the operators by the great advances in the technology and wide application of wood pulps as compared with the relatively neglected technology and limited use of agricultural pulps.

While a variety of reasons, all of them important, are given for the small use of residue plants, such as dirt, bulkiness, difficulties incident to their collection, transportation, and storage, etc., the most important reason probably is the feeling that yields of pulps are low, strengths are not high, and that clean pulps are costly to produce.

In the cases of esparto and seed flax tow, industry recognizes that better grades of papers for specific uses can be made from them than from wood pulps. In addition, such papers command a high-priced market. With these incentives, industry has attacked the problems connected with esparto grass and flax tow procurement with a will comparable with that found in solving like problems in harvesting hardwood species or pulpwoods from farm lots.

CLEAN PULPS

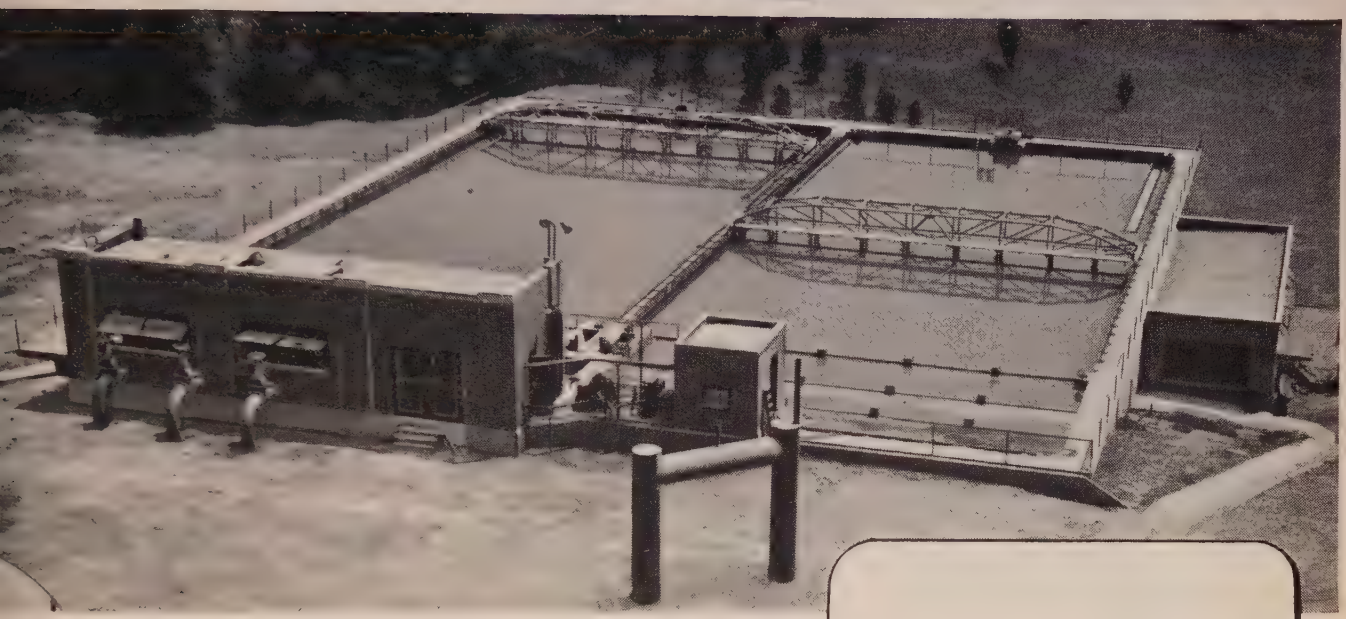
In the past, sufficient attention has not been devoted to the production of clean pulps. Straws contain, in addition to dirt, undesirable material such as nodes, heads, and grain kernels. In the development of the newer pulping methods for straw at the Northern Regional Research Laboratory, simple, effective methods in equipment standard in the pulp industry have been applied to the production of clean pulps. For example, in the mechano-chemical pulping process, the nodes, heads, and wheat grains are not pulped nor disintegrated. After washing the pulp, these may be removed by a simple riffling system prior to screening. The screenings and material from the riffler may then be further defibered by means of a machine operating on the general principal of the Hydrapulper. The defibered material is again riffled and screened. The rejects are discarded or used in making board or other pulp. Passing the screened pulp through a Vortrap, Dirtec, Hydraclone, or other dirt-removing machines is probably desirable before and after bleaching.

It has long been recognized that the pith cells in sugarcane bagasse, cornstalks, or other pith-bearing plants lower the

Table III. Agricultural Fibers and Pulpwoods; Variations in Fiber Dimensions and Ease of Pulping

Group	Fiber	Average length, mu	Average diameter, mu	Ratio: length to diameter	Relative density	Relative ease of pulping
1	Straws and esparto	1,100–1,500	9–13	110–120:1	Open	1
	Rice straw	1,450	8.5	170:1	Open	1
2	Stalks and reeds	1,000–1,800	8–20	80–120:1	Open	2
	Sugarcane fibers	1,700	20	85:1	Open	2
3	Woody stalks with bast fibers					
	A. Woody stems	200–300	10–11	<30:1	Dense	4
	B. Bast fibers	20,000–25,000	16–22	>500:1	Open	3
4	Leaf fibers	6,000–9,000	16–18	250–300:1	Open	3
5	Bamboos	2,700	14	200:1	Dense	4
6	Coniferous woods	2,700–3,600	32–43	75–90:1	Dense	4
7	Deciduous woods	1,000–1,600	38–50	<50:1	Dense	4

No Two Fingerprints Alike



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quality and raise the cost of making pulp from them. Very recently the Northern Regional Laboratory has developed a simple method for removing pith from sugarcane bagasse at the sugar mill. It is suggested that pith and blackstrap molasses, if mixed and sold as a feed base, will result in higher profits to the sugar industry. At the same time the process would provide a high-grade clean fiber at reasonable cost for making fine papers. This process not only removes the pith but the dirt and carbon present on the burned cane. Yields of unbleached strong fiber of more than 60%, based on pith-free fiber, have been produced from Florida bagasse by cooking with 15% caustic soda in the Hydrapulper for $1\frac{1}{4}$ hr.

While considerable progress has been made in preparing seed flax tow for use by the cigarette paper industry, there is still room for improvement in methods of removing the woody material from the tow. The woody stem requires much more severe conditions for pulping than the tow, so the tow because of the presence of the woody flax shives must be overcooked with a resulting much lower yield and quality of pulp than should be obtained.

High-Yield Specialty Pulps

It is most important in this era of great expansion in the pulp and paper industry and in the search for desirable pulp sources to redefine problems. In the first place, the problem concerns all countries and is of long rather than of short range, since the paper industry must expand as literacy, commerce, and standards of living advance. The viewpoint of producing substitutes from residues, tropical woods, hardwoods, or any other plant sources does not lead to industrial stability; only by finding the best combinations is permanence assured.

The examples mentioned show how problems specific to each pulp source may be solved. It must be recognized that pulping methods used for wood are probably not suitable as such for residues. Experience shows that, in general, alkaline chemicals produce the most satisfactory results. An attempt to duplicate the properties of wood pulps by pulping residues should not be considered. Rather, the effort should be directed to preparing pulp in such a manner that the unique properties inherent in its fiber dimensions and chemical properties result. This will require a certain amount of research, but the reservoir of knowledge on fundamental physical and chemical properties, pulping methods and machines, methods of screening, cleaning, refining, and paper machine operation contains the information, which with slight modifications, will solve most of the problems. The problems of mechanisms for collection, handling, and storage of plant fibers are not as difficult as those which required solution in the gathering of pulpwoods.

High-Yield Pulps

With emphasis placed on the use of residues to produce high-yield, high-quality, clean, specialty pulps, industry will awaken to the wide horizon of opportunities for new and improved products to which residues can contribute. Improved or new products signify higher prices and enlarged markets.

It has already been demonstrated with straw and sugarcane bagasse that very high-yield, high-strength pulps can be obtained by the newer procedures designed especially for pulping them. Over-all costs of chemicals, steam, and power are lower than for pulpwood, and these together with the high pulp yields bring production costs into line with softwood pulp.

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discussion in papermaking, it would be possible to predict this from their known intrinsic properties.

For example, tearing strength is known to be primarily dependent on fiber length. Poor formation in papers is due to the clotting or clumping together of fibers so that they are not uniformly distributed in the sheet of paper. Experience has demonstrated that long fibers or fibers which are fibrillated both contribute to poor paper formation. A combination of long fibers and highly fibrillated fibers produces the least desirable formation. Poorly formed papers do not possess a level surface and represent inferior sheets for printing and many other purposes.

In the poorly formed sheet the maximum strength which the individual fibers could contribute is not obtained because the clumps of fibers contribute very little to strength. Since paper strength is believed to be fairly related to the number of fiber bonds, it follows that fibers of a high ratio of length to diameter will, other things being equal, contribute greater strength than broader fibers of the same respective lengths.

Fibers with a high pentosan content hydrate or swell more rapidly on beating than those of low pentosan content. The drop in freeness of easily hydrated pulps is generally so rapid that very little if any fibrillation takes place in them during their refining for paper machine operation. Chemical pulps are generally less opaque than more highly lignified or mechanical wood pulps. Chemical pulps containing high amounts of pentosans are less opaque than those of lower pentosan content. Greaseproof or glassine papers with good formation may be made only from pulps high in pentosans.

Semichemical pulps high in pentosans produce stiffer and more crush-resistant papers than do pulps of low pentosan content. For this reason, wheat and rye straws and sugarcane bagasse are especially useful for the manufacture of corrugating papers. It is possible that respective blends of these pulps with semichemical hardwood pulps would be beneficial.

From the fibers in groups 1 and 2 (that is, straws, stalks, and reeds) it is possible to make excellent greaseproof and glassine papers so far as good transparency or resistance to grease and oil is concerned. The tear resistance of such papers might be improved by blends of softwood pulps and particularly pulps from bast, hard fibers, or bamboo if these were available and

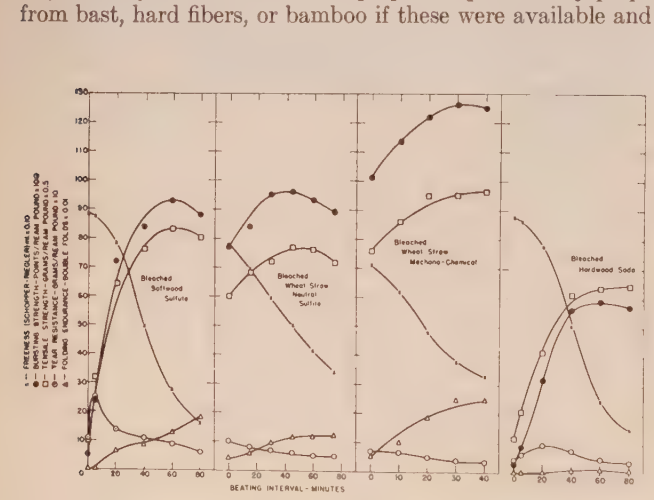

Fig. 1. Comparative strength properties of bleached wheat straw, neutral sulphite and mechano-chemical pulps, softwood sulphite pulp, and hardwood soda pulp

not too costly. Good writing or printing papers can be made wholly from such fibers. But except in papers from esparto, fillers are required generally to obtain the desired opacity. In our opinion, however, these pulps will find their greatest usefulness as blends with other pulps to produce superior types of papers.

The strength properties of bleached fine straw pulps produced by the two new high-yield processes developed by the Northern Regional Laboratory as compared with industrial samples of bleached softwood and hardwood pulps are shown in Fig. 1. The neutral sulphite straw pulp has a brightness of 70, a pentosan content of 30%, and a yield of 50% based on original dry straw, while the mechano-chemical pulp has a brightness of 82, a pentosan content of 33%, and a yield of 47%. The straw pulps exceed the wood pulps in both bursting and tensile strength at 500 Schopper-Riegler freeness, and they develop their maximum strength in about one half to two thirds the time required for the wood pulps. Also their maximum strength develops at a higher respective freeness. The mechano-chemical straw pulp shows higher folding endurance than either of the wood pulps, and the neutral sulphite pulp shows a higher value than the hardwood pulp.

The mechano-chemical pulp is the stronger of the two straw pulps. It contains more pentosans and shows a considerably more rapid strength development on beating. This pulp was used for machine runs on the experimental paper machine at the United States Forest Products Laboratory in the Spring of 1951. The neutral sulphite straw pulp is similar in properties to that used in making the industrial newsprint run at the Ontario Paper Co., Thorold, Ont. in the Spring of 1948. Pulps very similar in character may be obtained from depithed sugarcane bagasse fiber by the respective pulping processes.

These pulps, when blended in proper proportions with softwood pulps, will improve formation of the sheet, will improve the surface characteristics and printability, and will tend to reduce pick and fuzz. They are to be particularly recommended for use in uncoated papers such as writing and bond, tablet, ledger, mimeograph, and book. They are very useful in improving the smoothness and surface of supercalendered papers. They are also useful in coated papers and lithograph or label papers where a better coating surface is produced, and in waxing papers where better formation and greater transparency is obtained. They may replace part or all of the softwood sulphite in groundwood and magazine book papers where the addition of well-hydrated agricultural residue pulps not only increases strength and improves formation but also improves the normally limp sheet by adding the "snap" formerly present in magazine papers made wholly from chemical pulps. These pulps can replace the sulphite pulp used in newsprint manufacture. It is possible that a news sheet might be manufactured from waste deinked news plus a liberal percentage, e.g., 25% or more, of such pulps.

The foregoing statements are not just conjectures. They have been borne out in actual pulp and paper mill operations in many countries and in fairly large-scale experimental paper machine trials in this country. In Algeria, mixtures of straw and esparto pulps are used for fine paper manufacture in which high quality of product is maintained and costs of production somewhat lowered.

The use of the pulps from fibers of groups 1 and 2 will lead not only to improved paper products but will result in savings of power in beating and refining if the pulps are treated separately and blended just before final jordaning and the paper machine operation. There is also evidence that the use of these pulps in amounts up to 25 or 30% in blends with sulphite or kraft softwood pulps to make wrapping and bag paper may be justified in order to conserve softwood pulps. In that case, the wood pulp would be refined only to its maximum tear resistance and the residue pulps would be refined so that the final pulp blend would have the necessary freeness required on the paper machine. This is essentially the practice now used in Australia in making wrapping and bag papers from eucalyptus and imported softwood kraft pulps.

In general, the above statements are applicable to the use of bamboo pulps. Like esparto, bamboo pulps may be used 100% to manufacture a variety of papers. The long fibers adapt them for use in wrapping papers. It is suggested that blends of bamboo pulp with straw, sugarcane bagasse, or

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hardwood pulps might be suitable for wrappings and bags. This would be worth investigating.

It is well known that cigarette papers from bast fibers are highly preferred. In Spain rice straw is used for this purpose. It will be noted that rice straw fibers are of smaller diameter than any of the fibers in groups 1 and 2. Bast fibers have long been used for the manufacture of very high grade and expensive book, ledger, writing, condenser, and like papers which must resist handling.

United States currency paper contains seed flax tow pulp fibers because they increase tear and particularly scuff resistance. In the manufacture of certain book and writing papers made from bast fibers, blends of straw, bagasse, or other similar fibers could probably be used to advantage whereby costs might be reduced and quality maintained. The bast fibers must be reduced in length by beating and refining but their low pentosan and lignin contents and their narrow fiber diameter may be considered responsible for the very high qualities of the papers made from them.

The leaf or hard fibers, generally in the form of old rope, are used to produce our strongest papers. From the properties of the fibers their use might be predicted. They not only possess the fiber length necessary for the high tear resistance required in flour sacks, cement bags, and the like, but because of their narrow fiber diameter many more fibers are present in any area, all of which have to be broken to puncture the paper. Such fiber properties are also responsible for the high scuff resistance of the paper. Since the formation of the paper is superior to that obtainable with softwood pulps, the paper has a much more closed and smoother surface. The low lignin and medium pentosan content with high consequent cellulose content make also for ease of conversion to pulps having comparatively rapid drainage on the paper machine. Waste from the production of manila fiber is now being used for papermaking in Costa Rica. With improvements in recovering waste in hard fiber manufacture, low cost sources other than old rope should become available. Evidently, if costs permit, blends of agricultural or hardwood pulps with hard fiber pulps would produce good wrapping and bag papers. Blends of pulps from straw, stalks, or bast fibers with pulps from hard fibers should produce the strongest and the most tear-resistant very thin papers.

Papermaking is essentially concerned with the high-speed, continuous production of fibrous webs, now being put to use in thousands of ways. Of secondary concern are the raw materials used for pulping as long as quality products are produced at competitive prices. When raw materials are limited to a consideration of pulp production wholly or predominantly from wood fibers, as at present, much too narrow a view is taken of the possible expansion in the paper industry. The above discussion clearly shows that new and improved opportunities exist in the use of agricultural fibers as pulp sources not only to relieve pulp shortages now or in the near future, but that by judicious use of these fibers as blends with other fibers a wide horizon of new and improved papers is open to development by the industry.

METHODS FOR PRACTICAL UTILIZATION OF AGRICULTURAL RESIDUES IN MAKING INSULATING, HARD BOARD, BOXBOARD, AND SIMILAR PRODUCTS

Sugarcane bagasse has been used for 30 years to produce high-grade, rugged, insulating building materials in board form. Straw, cornstalks, flax shives, and licorice root have also been used successfully. Research at the Northern Regional Laboratory has shown that the rugged strength and high impact value of products made from such residues is due primarily to the incorporation in them of long fiber bundles. Such bundles can be produced from the above residues by mechanical means but they cannot be made from wood. This is due primarily to the difference in structure of wood and the various agricultural fibers.

Hard board has been successfully produced from sugarcane bagasse and from straw. Recently the Northern Regional Laboratory announced a process to produce from straw boxboard which can replace wood veneer in wire-bound shipping containers. Undoubtedly a large number of new uses in heretofore unexplored fields for the use of raw cellulose fibers will eventually be found. The agricultural residue fibers should be used where they will do a better job than may be done by wood fibers.

CONCLUSION

Agricultural fibers of diverse nature are readily available in most parts of the world. Research into their physical, chemical, and papermaking properties backed up with a considerable industrial experience in their use shows that they can greatly benefit the expanding paper industry not only by increasing pulp supplies but even more important, when properly used, to produce new and improved paper and board products. Now, industry is somewhat reluctantly extending the present limited use of these fibers. The greatest challenge to industry in this period of expansion lies in a reorientation in viewpoint so that raw materials are not confined to any particular type but are sought and used on the basis of producing better, newer, and more useful paper and board products. With such a reoriented viewpoint, the very great possibilities in the use of agricultural fibers become apparent. Also, with such a viewpoint, research and industry can proceed with confidence to solve the problems facing them.

Presented at the International Pulp and Paper Symposium under the auspices of the Wood Chemistry Subcommittee of the Food and Agriculture Organization, at The Institute of Paper Chemistry, Appleton, Wis., Sept. 17–21, 1951.

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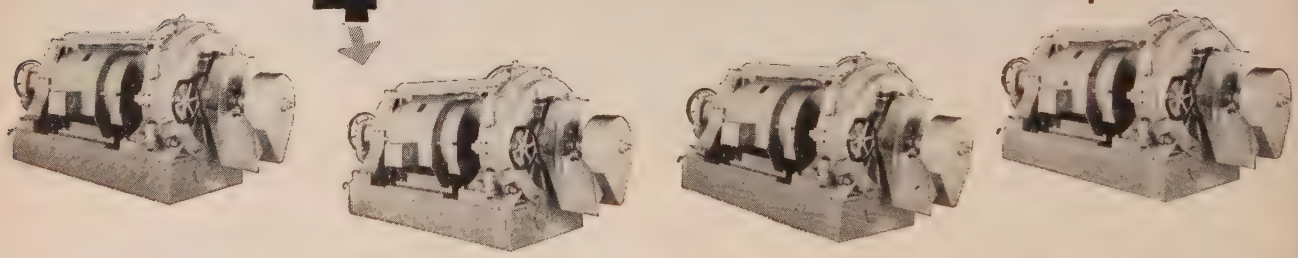

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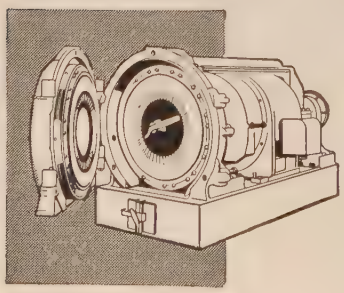

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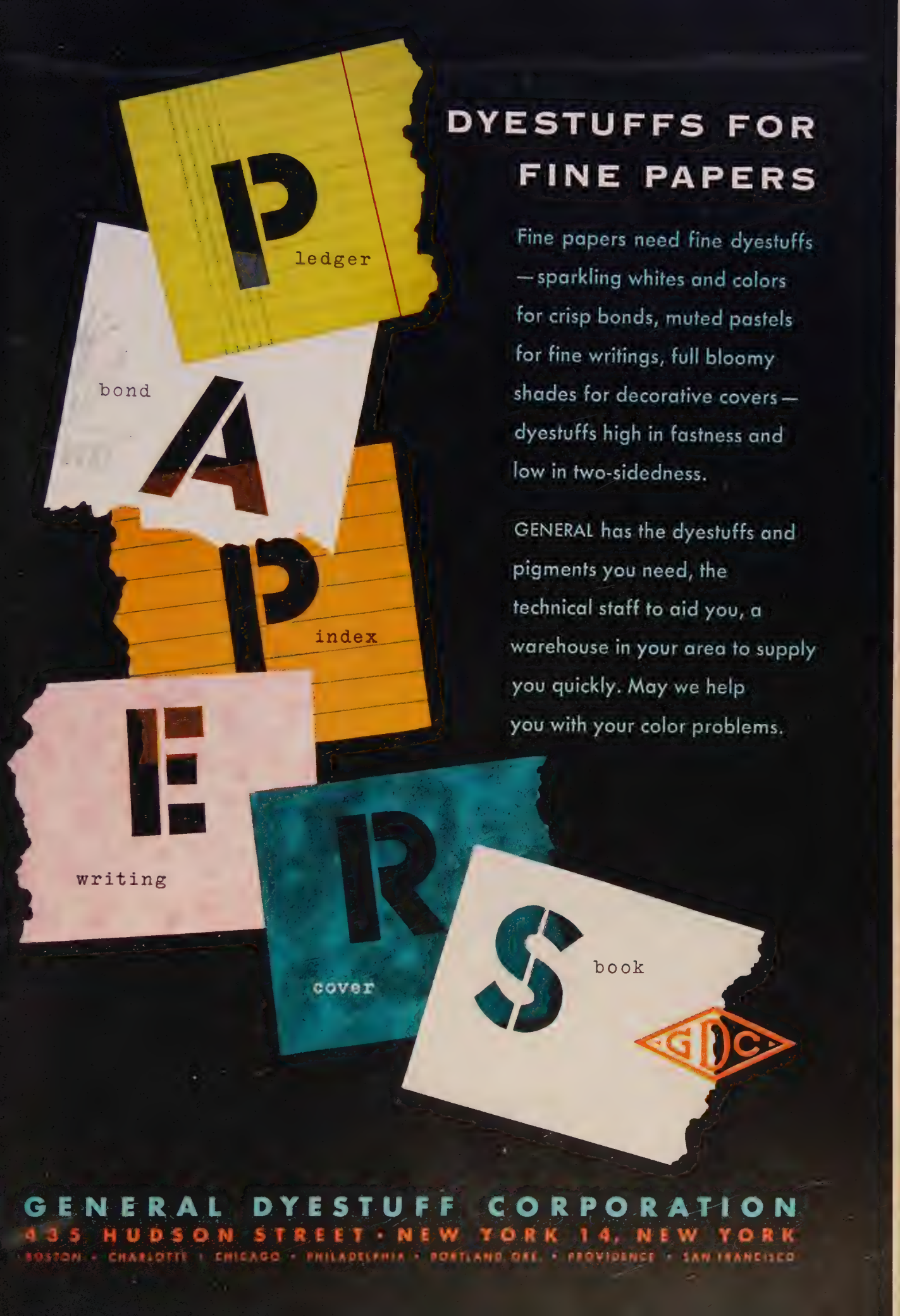

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The Present Status of the Chemistry of Cellulose

EMIL HEUSER

To BEGIN with the molecular structure, it is generally believed that the individual cellulose molecule consists of a linear chain of β-anhydroglucose units, linked together by 1,4-glycosidic linkages and that the cellulose fiber is built up of many such chains which, however, vary in length.

FASTER-SPLITTING BONDS

The generally accepted view is not in all points shared by Schulz and co-workers (*1–5*). In the early nineteen-forties, these investigators [possibly influenced by an earlier concept of Staudinger (*6*) on the presence of ester linkages in so-called "defective" celluloses] postulated that, first, in cotton and ramie celluloses, the chains as they originally exist in these plants are of uniform length, having a degree of polymerization (D.P.) of 3200 and, second, that celluloses of this type contain a small number of other than the normal glycosidic bonds, these bonds being weaker than the normal bonds and being regularly spaced along the chains at distances of 500 anhydroglucose units. The existence in cellulose of faster-splitting bonds could be derived from the fact that heterogeneous hydrolysis and oxidation of cellulose proceed at a considerably faster rate in the beginning than these reactions do later on. The concept of a regular spacing of special bonds (or so-called "weak spots") along the chain at distances of 500 anhydroglucose units resulted from the observation that the differential weight-distribution curves of cellulose samples which had been degraded to an average D.P. of 500 by acid hydrolysis in the heterogeneous system showed a certain accumulation of chains of such length. It was thought that this uniformity of the hydrolyzed sample at the 500 D.P. level was also reflected in the observation that the "nonuniformity quotient" (U) between weight-average molecular weight (M_w, as obtained by viscosity measurements and number-average molecular weight (M_w, as obtained by osmotic pressure measurements) showed a minimum at a D.P. of 500 when the U values of a series of progressively hydrolyzed samples were plotted against the D.P.'s of the samples.

Schulz and co-workers have drawn further support for their weak-linkages concept from a number of additional studies, both of their own and of others, among them a study of the homogeneous oxidation of cotton and ramie, dissolved in cuprammonium hydroxide, with air oxygen (*7, 8*); a study of the hydrolysis and oxidation of beech and spruce sulphite pulps, from which the conclusion was drawn that weaker bonds also occur in wood celluloses (*9*); a study of the acid hydrolysis of cotton under conditions in which undesirable influences of diffusion and slow penetration were eliminated (*10*); a study of the hydrolysis of cotton linters in which the chain-length distributions were derived from ultracentrifugal sedimentation (*11*); and a study of the heterogeneous oxidation of rayon pulp in which a very mild oxidating agent—tosyl chloroamide in very dilute solution—was used (*12*).

Support of the weak-bonds hypothesis was also drawn from the studies of Husemann, and Husemann and Carnap (*13, 14*), in which it was shown that slight mechanical treatment disintegrated the hydrolyzed fibers into short rodlike fibrillae with a pronounced accumulation of segments which measured 2250 A in length, corresponding to a D.P. of 450. Since the length of the fragments is in fair agreement with the (alleged) chemically determined 500-D.P. spacing of the chains, it was concluded that the faster-splitting bonds are arranged in the fiber perpendicular to its axis, and electron micrographs of the hydrolyzed fibers were interpreted to show such an arrangement (*5*).

The statement that, in celluloses of the cotton and ramie type, the chains are all of the same length (D.P. 3200) and, hence, that any polymolecularity which samples of these types may show results from degradation during isolation or other subsequent treatments, is not in agreement with the results obtained by other investigators. Thus, it was found (*15*) that cotton which was grown under careful exclusion of atmospheric influences and was taken from the boll shortly before opening is far from possessing a uniform chain-length distribution. Having an average D.P. of about 4000, the sample was composed of fractions ranging in D.P. from about 1450 to about 4300. It is thus seen that cotton possesses pronounced polymolecularity and it is very probable that the latter is to a great extent produced by nature. The same would seem to hold for the celluloses in wood pulps, although much of their greater polymolecularity results from the severer treatment required for their isolation (*15*).

As regards the regular spacing of the weak bonds at 500-D.P. distances, Jörgensen (*16a*) has shown that the accumulation of 500-D.P. material in cotton samples which had been hydrolyzed to an average D.P. of 500 is rather limited. In fact, fractionations of such samples showed that they contained varying but considerable percentages of widely varying D.P.'s. Likewise, the coincidence between an "accumulation" of 500-D.P. material and a minimum value for the nonuniformity quotient, as claimed by Schulz, could not be confirmed. Instead, the U values were found to decrease with progressive degradation without passing through a minimum.

Doubts as to the existence in cellulose of regularly spaced weaker bonds were expressed also by Emery and Cohen (*17*) and by Matzusaki and Sobue (*18*), and indication for their existence could not be derived from

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the results of hydrolysis either with acids (*52, 53*) or with enzymes (*19*).

EFFECT OF AMORPHOUS TO CRYSTALLINE RATIO UPON THE RATES OE REACTIONS

It would seem to be extremely difficult to furnish convincing proof for the presence of faster-splitting bonds since, in cellulose, we are dealing with a system of chain molecules in which amorphous (i.e., easily accessible portions) alternate with crystalline portions (i.e., of much reduced accessibility). It is well known that this duality in the fine structure of the cellulose fiber governs the course of many cellulose reactions (*20*) and may be assumed to determine the rate of these reactions to a far greater extent than could be expected from the presence of a relatively very small number of faster-splitting bonds. At any rate, the faster-splitting bonds should still be discoverable when cellulose is degraded in the homogeneous system in which the duality of the fine structure is eliminated or much reduced. The results of the hydrolysis of cotton cellulose dissolved in phosphoric acid give no indication for an initially faster rate (*16a*). The results obtained with wood celluloses are still somewhat erratic and need further clarification. This would also seem to hold as regards the oxidative degradation of cellulose in cuprammonium solution, for which Schulz (*7*) found an initially faster rate.

The lack of clarity suggests that studies on the rate of degradation of cellulose with the aim of detecting faster-splitting bonds should, nevertheless, be carried out in a homogenous system only, and perhaps best in phosphoric acid solution, in which the accessibility of the chains is considerably increased (*21, 22*) and complications as they exist in the cuprammonium (and cupriethylenediamine) system are absent. Furthermore, the starting cellulosic material should preferably not be fibrous cellulose but a modification in which the complete amorphous state is approached as closely as possible (*20*). Such modifications may be obtained by dry grinding of cellulosic materials in a vibratory (oscillating) mill, which was described by Hermans and Weidinger (*23*), Steurer and Katheder (*24*) and Forziati, Stone, Rowen, and Appel (*25*). Reduction of crystallinity, although to a less far-reaching degree, has also been accomplished by the action of swelling agents, such as quaternary ammonium bases and alkylamines, both of which were used by Segal, Nelson, and Conrad (*26*).

THE NATURE OF THE HYPOTHETICAL FASTER-SPLITTING BONDS

If the weaker bonds actually exist, the question of their nature arises. From earlier studies, the probability that these bonds are of the α-configuration is very remote. The weak bonds could then simply be normal β-1,4-glycosidic linkages located in the neighborhood of certain foreign groups (e.g., carboxyl or carbonyl groups), for which reason they would be more easily attacked by degrading agents than the rest of the glycosidic bonds. However, this assumption would not explain their periodicity.

In wood pulps the weak bonds could exist at points at which possibly anhydroxylose or anhydromannose units (*16a*) are built into the anhydroglucose chains. The occurrence of such units as part of the regular chains has been inferred from the difficulty of isolating cellulose from wood (and similar incrusted sources) completely free of xylan and mannan. More recent investigations by Wise, *et al.* (*27*), Meller (*28*), and Hass (*29*) would seem to lend support to this interpretation.

The weak bonds have also been referred to as cross linkages between individual chains by Pacsu and coworkers (*30*). Pacsu's much discussed cellulose formula, in which anhydroglucose chains are cross linked to cellobiose units through acetal bonds at regular distances, has been criticized by many (*31–36*). Today this formula appears to be no longer defended.

From time to time carboxyl groups, which are found in many cellulosic materials, have been looked upon as fundamental constituents of the molecular structure of cellulose. It would seem that this concept can now be definitely abandoned in the light of results brought forward in more recent studies. Thus, Meyer and coworkers (*33, 37*) found that both cotton and alpha-cellulose (from wood pulps) in the form of precipitates from their cuprammonium solutions, carefully washed, acidified, and electrodialyzed, are free of carboxyl. Any acidity which may be detected in such purified preparations by using Neale's method of titrating with alkali in the presence of sodium chloride (or similar techniques) is simply a result of the accumulation in cellulose of hydroxyl groups. Similar conclusions were reached by Ant-Wuorinen (*38*). "Pure cellulose" is thus looked upon (*33*) as a β-1,4-polyglucosan, analogous to and isomeric with amylose, an α-1,4-polyglucosan. The methods of determining carboxyl in cellulosic materials and oxycelluloses were improved by various investigators (*39–42*).

ACETOLYSIS AND OLIGOSACCHARIDES

The β-1,4-anhydroglucose chain structure of celluloses has received new support from recent results of acetolysis. By using chromatographic techniques, Dickey and Wolfrom (*43*) resolved the acetolyzate of cellulose into a polymer-homologous series of crystalline sugar acetates, comprising all members from glucose to cellohexaose. Through this study, the doubt which had existed for some time with regard to the identity of the cellohexaose among the oligosaccharides from cellulose has now been removed.*

HYDROLYSIS AND FINE STRUCTURE

The hydrolysis of cellulose with mineral acids has frequently been referred to earlier in this review. It may be added that, in recent years, this reaction has primarily been used to clarify our concepts on the fine structure of cellulose (*32*). More recent investigations referring to the phenomenon of recrystallization (*44, 45*) and the concept of "limiting" hydrolysis as a means of separating the amorphous from the crystalline portion (*46, 47*), were reviewed in the beginning of 1950 and 1952 (*48*). The earlier review also contains references to and a discussion of other chemical, as well as of the physical, methods for determining the crystalline-amorphous ratio in cellulosic materials. Other investigations in which limiting hydrolysis and recrystallization play essential parts were published by Jörgensen

* According to a private communication from Dr. Wolfrom, all sugars have been obtained in a free form and in the meantime another oligosaccharide, the celloheptaose, has been added.

(*49*), Battista (*50*), Morehead (*51*), and by Rånby and Rånby and co-workers (*52–55*). In interpreting the results obtained in these studies, Rånby has gone farthest—i.e., by suggesting that the fragments which may be obtained by limiting hydrolysis and with the aid of ultrasonic treatment, when viewed under the electron microscope, might represent the micelles, the hypothetical existence of which had long been derived from x-ray analysis.† Others (*48, 56*) look upon these fragments as incidental rather than fundamental units of the cellulose fiber.

MICROBIOLOGICAL (ENZYMIC) HYDROLYSIS

As regards the microbiological (enzymic) hydrolysis of cellulose, earlier hypotheses and the results from which the hypotheses were drawn have been temporarily questioned (*57*), postulating that no intermediates, such as cellobiose, are formed and that the only product resulting from this type of degradation is glucose. Further studies in the same laboratory (*58*) confirmed, however, that cellobiose does appear and does play an essential part in the enzymic decomposition of cellulose.

What is still in a state of dispute is the mode of microbiological attack. Siu (*57*) and Siu and co-workers (*59*), as well as Greathouse (*60*), have interpreted their results as being those of a random attack—i.e., a cleavage of glycosidic linkages at any point along the chain, whereas Walseth (*61*) concluded that the enzyme attacks the chains in an endwise fashion.‡ Obviously, more experimental work would be necessary for a clarification of this question. On the other hand, agreement seems to exist as to the preference which the cellulolytic enzymes display for regenerated cellulose and for chain molecules located within the amorphous portions of cellulosic materials (*59–62*). In fact, this preference may be utilized for a separation of the amorphous from the crystalline portion, the result of which, however, differs from that which is obtained by hydrolysis with acids (*61*). Whereas, in the latter process, the average D.P. of the residue had decreased to a very low level, enzymic hydrolysis (using fungi or extracts therefrom) left a residue which showed practically the same average D.P. as the starting material (*61*). On the other hand, certain lignin-destroying fungi, such as *Polyporus anceps* and *P. paragamenus*, which were used in an attempt to isolate the cellulose constituent from some hardwood species, although removing most of the lignin, did lower the D.P. of the residue considerably (*63*).

Our knowledge on the resistance of cellulose derivatives toward microbiological attack has been widened in various respects. A considerable variety of ester and and ether substituents exert a positive effect by causing one or more of the three hydroxyl groups per anhydroglucose unit to be blocked (*60*). More precisely, only one of the three hydroxyl groups need be substituted to secure immunity against microbiological attack (*57, 59*). If, on the other hand, the degree of substitution remains below one, the attack becomes positive. In contrast, the results obtained with certain fully substituted simple sugars would seem to differ with the type of microorganism used. Thus, Greathouse (*60*) and Siu (*59*) found cellobiose octaacetate and pentaacetylglucose completely resistant to the growth of the fungi *Myrothecium verrucaria* and *Chaetomium globosum*, whereas Abrams (*64*) reported that one of these fungi (*C. globosum*) attacked these sugar derivatives very strongly. In addition, Abrams found that native cotton cellulose fiber was completely resistant to the enzyme extracts from *C. globosum*.

MOLECULAR WEIGHT

Further clarification of the discrepancy between molecular weight values obtained by ultracentrifugal techniques on the one hand and viscosity and osmotic pressure measurements on the other (*65, 5b*) has not progressed very much since this subject was last reviewed by the writer (*66*).

More progress has been made as far as the viscosity-molecular weight relationship is concerned. Mark (*67*) has once more reviewed this relationship, with particular consideration of the limitations of the validity range of the Staudinger function and has shown that these limitations, which start to affect values above a D.P. of about 1000, are the result not only of the size and the effective shape of the long-chain molecules but also of the complications which arise from the movement of the liquid through, as well as around, the chain molecules. The effect of cellulose concentration upon cuprammonium viscosity, particularly for the range of relative viscosities between 1.5 and 10, was the subject of a study by Calvert and Clibbens (*68*); these authors also gave a critical review of pertinent equations, such as those suggested by Baker, Farrow and Neale, de Jong, Kruyt, and Lens, Schulz, and Blaschke, and Martin. Although all these equations appear to be satisfactory, as far as relative viscosities are concerned, the equation of de Jong, Kruyt, and Lens and Martin's modification of the latter would seem to be preferable when intrinsic viscosities are to be calculated. Studies with similar objectives were made by Parisot and Cyrot (*69*), by Valtasaari (*70*), and by Ivanov, Golova, and Zakharov (*71*). Two of these authors also made a comparative study of viscosity molecular weights and those obtained by ultracentrifugal sedimentation and diffusion techniques (*72*). The importance of considering the influence of the rate of shear upon intrinsic viscosities, both in cuprammonium hydroxide and in cupriethylenediamine solution, in cases in which the D.P.'s are above 1000, was stressed by Conrad, Tripp, and Mares (*73*) and by Wilson (*74*). A subcommittee of the German Association of Pulp and Paper Chemists and Engineers issued a standard method for D.P. determinations of cellulosic materials by the Staudinger cuprammonium method (*75*). The undesirable effect of air oxygen would seem to have been greatly reduced as the result of two recent studies on the effect of adding to the solution either cuprous chloride and (or) copper wire (*76*) or copper wire alone (*77*). The cupriethylenediamine method has received increasing attention and seems to gain preference over the cuprammonium hydroxide method (*78–80*).

A substantial portion of the Cellulose Division meeting at Boston in April, 1951, was devoted to the viscosity-molecular weight relationship (*81*), and the chairman of the Viscosity Subcommittee, A. F. Martin,

† Rånby was more positive in his interpretation of the fragments (from hydrolysis combined with ultrasonic waves) as "micelles" in his earlier publications [see, e.g., Rånby and Ribi (*52b, 52d, 53*)].

‡ It should be added here that Greathouse (*60*) has interpreted Walseth's results (from the latter's doctoral dissertation) in favor of a random attack. See also the interpretation of the results by Bauer and Pacsu (*16b*, p. 393).

made several recommendations for simplifying the cuprammonium and cupriethylenediamine techniques and for expressing the results with the aim of eventually establishing a standard referee method. His recommendations were based upon an earlier publication of his own (*82*).

Jayme (*83*) succeeded in what had been tried earlier in vain by Traube (*84*)—namely, in replacing the copper in cupriethylenediamine by cobalt and in dissolving (up to 7%) cellulose in the new (cobaltiethylenediamine) reagent. A deep wine-red, highly viscous solution is obtained. Jayme assumes that the mechanism involved is analogous to the formation of the cellulose cupriethylenediamine complex. In this connection it is of interest to note that, as a result of the studies by Reeves on the action of cuprammonium hydroxide upon methyl glucosides (*85*) and on the formation of a mannosan cuprammonium complex (*86*), we might have to revise the concepts hitherto held with regard to these mechanisms. According to Reeves, the cellulosecuprammonium complex may possibly be looked upon as a simple bimolecular association of the cuprammonium radical with the 2,3-glycol grouping in the anhydroglucose unit.

Some items of special interest pertaining to the viscosity of cellulose derivatives may be inserted here. The objections which some investigators have expressed against Staudinger's practice of using the viscosities of cellulose acetates in cuprammonium hydroxide solution for D.P. determinations without first removing the acetyl groups were definitely disposed of by the results of a study by Cox and Battista (*87*, *88*). Thus, the advantages of using only one solvent (cuprammonium hydroxide) may well be realized. In addition, such cellulose acetates are "eligible" for the application of this technique which, because of their low acetyl content, are insoluble in organic solvents.

The interesting and long known, but not fully understood, phenomenon of the "salt effect"—i.e., a viscosity increase in cellulose acetate solutions—which is caused by reactions of (bivalent) salts with both carboxyl and bound-sulphate groups in the acetate, was the subject of a comprehensive study by Malm *et al.* (*89*). In a subsequent, equally extensive study on the effect of bound metal in ethylcellulose solutions, Evans and Spurlin (*90*) showed that the salt effect affects the osmotic pressure as well, lowering it as the viscosity is increased.

The phenomenon of viscosity decrease in cellulose xanthate solutions as a function of electrolyte concentration (*91*) was the subject of a new study by Tait, Vetter, Swanson, and Debye (*92*). In addition, light scattering measurements, as well as data on the variation of intrinsic viscosity with molecular weight in various solvents, showed that the change in intrinsic viscosity with electrolyte concentration could be accounted for by a change in configuration or degree of coiling of the molecule. Calculations indicated that, in pure water, cellulose xanthate should approach a rigid rod configuration, whereas in 6% caustic soda solution coiling occurs and the molecule resembles more a random configuration.

As regards the other methods for determining the molecular weight of cellulose, such as the ultracentrifugal techniques and the osmotic pressure and light scattering measurements—apart from what has just been said—numerous studies of recent years were concerned with improving technical details and modes of evaluation of the data (*93*, *94*). These methods were also discussed at the Boston meeting (*95*), where it was stated by Doty that, only six years after its appearance, the light scattering method may now be regarded as a firmly established means for determining the weight, the size, and the activity coefficient of macromolecules in solution.

END-GROUP ASSAY

Some interesting work has been done in recent years on molecular weight determination by end-group assay. The liberation of formic acid from starch, cellulose, and other carbohydrates by the action of periodate, suggested as an end-group assay method by Hirst and coworkers in 1945 and subsequently discussed by Percival (*96*), was used by Percival and Ross (*97*) for determining the D.P. of cellulose from marine algae. The D.P. found was 160, which, however, indicates degradation on isolation. It would seem that no comparison of this end-group assay with other end-group methods or with other molecular weight determination methods has as yet been made. Whether the reduction of 3,5-dinitrosalicylic acid in alkaline solution and the colorimetric estimation of the resulting nitroaminosalicylic acid, which was successful with amylose and lichenin (*98*), has in the meantime been tried on cellulose, has not yet become known.

After Militzer (*99*) among others had shown how the Kiliani cyanohydrin reaction may be used as a quantitative measure of the reducing power of simple sugars, as well as of di- and trisaccharides, similar techniques were suggested for determining the end groups in polysaccharides. Thus, Yundt (*100a*) used a modified technique for determining the average chain length of a xylan preparation. The D.P. thus obtained checked well with the osmotic pressure D.P. (of a methyl derivative of the xylan) of about 39 (*100b*). Yundt's data on the application of the modified technique to "standard" cellulose" (D.P.'s from 2000 to 3000) give no details (*100c*). Isbell (*101*) has reported that the reducing end groups of polysaccharides may be determined by using cyanide in which the carbon atom is labeled with the radioactive C^{14} isotope without thus far having published details of his study. However, Frampton, Foley, Smith, and Malone (*102*) also applied a modified cyanohydrin assay technique, similar to that used by Yundt. They have given full details of a study made on (dewaxed) cotton cellulose without, however, expressing their results in molecular weights or D.P.'s. They also followed the hydrolysis of cellulose (a sample of Whatman filter paper) with hydrochloric acid by using the new method. The D.P.'s thus obtained by end-group determination were compared with the viscosity D.P.'s determined according to the cupriethylenediamine method as some years ago applied by Staudinger and Daumiller. The agreement was satisfactory considering that the end-group D.P.'s represent number average molecular weights, whereas the viscosity D.P.'s are weight-average molecular weights.

FRACTIONATION AND CHAIN-LENGTH DISTRIBUTION

For the purpose of subdividing a cellulosic materia

into its portions of varying chain length, the so-called nitrate method is widely used. This method comprises conversion of the cellulose sample into a nitrocellulose preparation, determining the viscosity of the latter in an organic solvent for obtaining its average D.P., fractionating the solution and determining the D.P.'s and percentage weights of the individual fractions and drawing chain-length distribution curves from the latter two data. Although many improvements as to experimental details of the individual steps and as to interpretation of data were made in recent years, including some interesting and more radical modifications of some of the individual steps (*103–110*), some items remain to be clarified. Among these are the variation in nitrate yield, the removal of noncellulosic carbohydrate nitrates, and the extent to which the latter influence the results. Another item concerns the decrease in viscosity which the nitrate suffers while standing in acetone (or other organic solvents). Various investigators have directed attention to this phenomenon and how to eliminate it. Recently Emery and Cohen (*17, 111*) and Campbell and Johnson (*112*) have suggested that the initial rapid drop in viscosity is possibly caused by a breakdown of hydrogen bonds between adjacent chain molecules, whereas the subsequent slow decrease might result from a cleavage of (normal) glycosidic linkages in the chain. Attempts to standardize the experimental details of the nitration procedure, which are now under way (*81*), will undoubtedly consider the results of these studies and the suggestions made therein for remedying the sources of error.

As regards the fractionation step, fractional solution and fractional precipitation were made the subject of a systematic comparative study, which showed that fractional precipitation results in a more complete subdivision of the sample and should, therefore, be given preference (*113*). However, it should be added here that the last word will have to be left to the recommendations of the Subcommittee on Chain-Length Distribution of the Cellulose Division which is investigating the question of standardization under the chairmanship of R. L. Mitchell (*81*).§

An altogether different method of fractionation has been suggested by Brooks and Badger (*114*). It is based upon the ability of starch to adsorb cellulose nitrate from a solution in acetone admixed with several cosolvents. The extent of adsorption has been found to increase with the molecular weight of the nitrate, and fractionation was accomplished by elution chromatography (with starch as the adsorbent).

Progress has also been made with regard to the fractionation of other cellulose derivatives. Thus, Scherer and McNeer (*115*) have worked out precise fractionation techniques, suitable for the subdivision of large quantity samples, for ethylcellulose and Scherer and Thompson (*116*) did the same for secondary cellulose acetates. Timell and Purves (*117*) applied the nitrate method to partially and low-methylated cellulose samples (0.1 to 0.6 methoxyl group per anhydroglucose unit) taken at intervals during the early stages of methylation. Of the fractions thus obtained, both D.P.'s and the degrees of methylation were determined. It is interesting to note that, as could have been anticipated, methylation was found to take place principally within the amorphous regions of the samples and that the shorter chains were methylated at a considerably faster rate than were the longer ones.

§ Attention is also directed to the symposium on molecular weight determination methods at the Atlantic City ACS meeting (Cellulose Chemistry Division) in Sept., 1952, where the attempts to standardize the nitrate method were continued.

POLYMOLECULARITY AND PHYSICAL PROPERTIES

The results of more recent studies on the effect of the polymolecularity of cellulose derivatives (or celluloses regenerated therefrom) upon their physical properties have, in general, confirmed the views hitherto held—namely, that there are limits to both the short and the long-chain materials which may be present and still yield products of desirable properties. Too much short-chain material is undesirable because tensile strength and folding endurance suffer, and too much long-chain material causes difficulties in certain operations—for example, in the filtering of viscose and its spinning into rayon. The relation between the quality of viscose rayon and its polymolecularity was the subject of various investigations. Mitchell (*118*) studied the effect of the various viscose manufacturing steps—particularily, the aging of the alkali cellulose—upon chain length and chain-length distribution of the resulting intermediates and of the final product. Coppick, Battista, and Lytton (*105*) demonstrated, by way of their summative method of fractional analysis, the pronounced effect which the chain-length distribution of the original pulp has upon the chain-length distribution of the rayon made therefrom, and Davis (*119*) showed how the fatigue resistance of rayon tire cord correlates with the amount of low-D.P. material contained in the rayons and the corresponding pulps.*

In contrast to the generally accepted view—which appears to be confirmed by the results of the above-mentioned studies (*118, 105, 119*)—are the conclusions which were drawn by Scherer and Rouse (*121*), who stated that the presence even of large amounts of low-D.P. material (below a D.P. of about 100) in a blend of nitrocellulose fractions of varying D.P.'s has relatively little effect upon the physical properties of the films prepared from such blends, and by Scherer and McNeer (*122*) who found that, in the case of ethylcellulose, the short-chain material even improved the mechanical film properties of the blends, as compared with the original ethylcellulose films.

CELLULOSE DERIVATIVES

The versatility of cellulose reactions which are mainly those of the hydroxyl groups in cellulose, is enormous. Ott (*123*) has discussed this versatility from various angles and has illustrated the profound influence which the nature, size, and distribution of the substituent, as well as the degree of substitution, exert upon such properties of the derivatives as moisture sensitivity, softening point, tensile strength, solubility and compatibility with plasticizers.

As regards new cellulose derivatives, interesting, and useful esters and ethers have been prepared and numer-

* In his paper before the Appleton Symposium, Sept., 1951, Haas (*120*) expressed the opinion (derived from experimental studies) that the degradation which wood pulp suffers on aging of its alkali cellulose removes all original chain-length distribution differences and that, hence, the chain-length distribution would seem not to be an important characteristic for viscose pulps that have to undergo alkaline aging. It would seem that the difference in views, as expressed by the various investigators, needs further clarification.

ous experimental studies on the distribution and position of the substituents and on the rate of the substitution reactions have contributed much to our present-day fundamental knowledge. Of the numerous studies, we should like to direct attention to the following: on methyl- and ethylcelluloses by Timell (*124*); on hydroxyethylcellulose by Cohen and Haas (*125*); on cellulose xanthates by Scherer and Phillips (*126*); on the determination of primary hydroxyl groups in secondary cellulose acetates by applying the methods of tosylation (with subsequent iodination) and of tritylation by Malm, *et al.* (*127*); on the rate of tritylation and tosylation of cellulose by Honeyman (*128*) and by Heuser, Heath, and Shockley (*129*); on carboxymethylcellulose by Reid and Daul (*130*) and by Rydholm (*131*); on cellulose thiocyanate by Carson and Maclay (*132*): on a homologous series of aliphatic acid esters from the acetate through the palmitate by Malm, *et al.* (*133*); on the action of diazomethane on cotton cellulose and modifications of the latter by Head (*134*); and on cellulose phosphates by Reid and Mazzeno (*135*). We also should like to direct attention to a discussion of some new concepts concerning the structure of cellulose nitrates and acetates by Happey (*136*).

ALKALI CELLULOSE

It would seem that the much-disputed question as to whether alkali cellulose is a chemical compound or the result of a purely physical adsorption of aqueous alkali on cellulose has not yet found a final answer. Among the studies which concerned themselves with this and related questions in recent years, we may mention a study by Jäger (*137*), dealing with an analysis of the theoretical considerations underlying the methods of Vieweg, Schwarzkopf, and Coward and Spencer; a study by Champetier and Néel (*138*) and by Champetier and Ashar (*139*) as parts of a general series of investigations on addition reactions and the formation of molecular compounds in heterogeneous "polymer-binary" liquid systems; a study by Calkin (*140*) on the determination of the ionization constant of cellulose in the cellulose-sodium hydroxide-water system; by Lauer (*141*, *142*) on the question of which of the three hydroxyl groups in the anhydroglucose unit of the cellulose chain are involved in the compound formation with sodium hydroxide, and a study by Samuelson, Gärtner, and Johansson (*143*) on the exchange in alkali cellulose of Na^{23} for the radioactive isotope Na^{24}.

OXIDATION

The fact that the cellulose chain molecule may be attacked by oxidizing agents at six different points (and at seven, if faster-splitting bonds were present) explains the great versatility of its oxidation reactions. Although some advances as regards the elucidation of the mechanisms involved in these reactions have been made, much remains to be investigated.

Periodate and chlorous acid oxidations were combined by Head (*144*) to produce from Kenyon's (*145*) nitrogen dioxide oxycellulose, a tricarboxylic acid cellulose which, on hydrolysis, was cleaved into glyoxylic and *meso*-tartaric acids. It was concluded that, by this series of reactions, the molecular structure of the nitrogen dioxide oxycellulose appears confirmed. However, it would seem that this conclusion does not convey the whole story. For Nevell (*146*) has shown that, in the nitrogen dioxide oxycellulose, only 85% of the carboxyl groups (determined by the Tollens-LeFèvre method) were of the uronic acid type, the rest being in other than the 6-positions, probably in the 2- and 3-positions. In addition, Nevell has stated that the alkali sensitiyity of the nitrogen dioxide oxycellulose must be attributed to the newly formed reducing groups rather than to the carboxyl groups. It remains to be seen how these results fit into the picture which we have derived for the nitrogen dioxide oxycellulose from Kenyon's and from Maurer's work. Information on the formation of reducing groups (and of a corresponding decrease in D.P.), which occurs while the nitrogen dioxide oxycellulose is being prepared may be found in an extensive publication by Pfeiffer and Krüger (*147*). In addition, attention is directed to the studies of Rogovin and co-workers (*148*, *149*), who investigated various properties of the tricarboxylic acid cellulose (see Head, above).

The instability of periodate oxycelluose toward aqueous alkali was the subject of various studies, among them one by Head (*150*), who demonstrated that the mechanism involved is a straightforward alkaline hydrolysis of glycosidic linkages. Periodate oxycellulose may be stabilized toward alkali attack by converting its dialdehyde grouping into a dicarboxylic acid grouping through treatment with chlorous acid, as was shown by Davidson and Standing (*151*) and by Meller (*152*). Davidson demonstrated that this stabilization is limited, however, for when the stabilized periodate oxycellulose was allowed to stand in water at 90°C. for some time, carboxyl was cleaved off and the thus ionized carboxyl, in turn, catalyzed the cleavage of glycosidic linkages, as indicated by a decrease in viscosity. Meller subjected other oxycelluloses, such as chromic acid and calcium hypochlorite oxycelluloses, to the stabilization treatment with chlorous acid and, in addition, used this treatment for the oxidation of hydrocellulose. He termed the resulting oxycelluloses "gluconic-acid oxycelluloses," assuming that they contain gluconic acid terminal groups, an assumption which has now been proven by Blair and Reeves (*153*).

Practical application of the stabilization with chlorous acid was made by Reeves and Darby (*154*), who converted a sensitive periodate-oxidized cotton thread into a stronger and more alkali-resistant product. With hypochlorite-oxidized (bleached) cotton fiber, the chlorous acid treatment was not effective, but treatment with ethereal diazomethane (according to an earlier suggested procedure) resulted in the desired stabilization effect (*155*). The different behavior of the two oxycelluloses toward the two stabilization agents may probably be explained on the assumption that bleaching with hypochlorite produces carboxyl groups in the 6-positions. These remain indifferent toward subsequent treatment with chlorous acid, but are converted into methyl ester groups by the diazomethane treatment. In the periodate-oxidized fiber, the unstable dialdehyde grouping is changed into the stable grouping by either treatment, although the mechanism involved in the reaction of diazomethane with the dialdehyde grouping in periodate oxycellulose needs further study (*134*, *156*).

The aging of alkali cellulose, which is practiced in the viscose-making process prior to xanthation, is brought about by the action of air oxygen. A clarification of the mechanism involved has occupied cellulose chemists for a long time but has not been accomplished in a manner which would be satisfactory in every respect. Haskins and Hogsed (*157*) have recently studied the aging process, using hydrogen peroxide instead of air oxygen for oxidation and following the oxidation by hydrolysis of the oxidized cellulose, in an attempt to find the fragments which represent the end groups. They isolated small amounts of arabonic acid from the aged alkali cellulose and saw in the formation of this acid the clue to a new hypothesis for explaining the mechanism involved. This mechanism is consistent with the behavior of β-alkoxyl-carbonyl compounds in alkali but would seem to need further clarification. Other mechanisms have been proposed, such as that of Sönnerskog (*158*), who has invoked the theory of ester resonance in a mechanism similar to that proposed by Heuser and Haskins some time ago (*159*) and by Entwistle, Cole, and Wooding (*160*), who regarded the aging of alkali cellulose as a chain mechanism of autoxidation, which involves the appearance of transient free radicals. It remains to be seen to what extent the various new hypotheses can be reconciled with one another and, further, whether alkaline hydrogen peroxide oxidation is actually equivalent to the aging of alkali cellulose in the presence of air oxygen.

REDUCTIC ACID FROM OXYCELLULOSES

Upon acid hydrolysis of certain oxycelluloses, Sohn (*161*) obtained small but nevertheless significant amounts of reductic acid. The molecular structure of this acid has a certain similarity to a portion of the ascorbic acid (vitamin C) structure. The formation of reductic acid from oxycellulose would seem to rest upon the presence (or formation) of glucuronic acid groups and to involve the subsequent reactions of decarboxylation and dehydration of the glucuronic acid units. In fact, the yields of reductic acid were greatest when oxidants, such as chlorine in water, nitrogen dioxide, permanganate and bichromate in the acid medium were used which preferably attack the hydroxyl groups in the 6-positions and which simultaneously bring about cleavage to shorter chains. Accordingly, the yield of reductic acid was considerably smaller with periodate and hydrogen peroxide oxidations. This latter observation is interpreted to show that hydrogen peroxide also preferably attacks the hydroxyl groups in the 2- and 3-positions.

The formation of reductic acid on subsequent decarboxylation and dehydration of glucuronic acid groups suggests a relation to the formation of furfural from certain oxycelluloses. In fact, the formation of reductic acid from oxycelluloses is always accompanied by the liberation of furfural and the oxycelluloses which Sohn found to give the highest yields of reductic acid are those which also give the highest furfural yields (*162*). The reaction may be expressed thus:

$$C_6H_{10}O_5 \rightarrow \begin{cases} C_5H_4O_2 + CO_2 + 3H_2O \\ \text{furfural} \\ C_6H_6O_3 + CO_2 + 2H_2O \end{cases}$$

Glucuronic acid — Reductic acid

The nature of the intermediates of the two reactions is still a matter of speculation. Likewise, still unexplained remains the observation that certain, though rather limited, amounts of reductic acid were obtained from the hydrolyzates of unoxidized celluloses. As an example, cotton yielded 0.325, cotton linters 0.051, and spruce sulphite pulp 0.154% reductic acid, whereas, when oxidized with chlorine water, cotton linters gave 2.02 and sulphite pulp 2.06%. The greater yields of reductic acid from the acid-type oxycelluloses suggested to Sohn a mechanism which is based on the hypothesis of Staudinger and Roos (*163*), postulating the formation of unsaturated groupings as intermediates in the formation of reductic acid.

USE OF RADIOACTIVE ISOTOPES

In closing, reference may be made once more to the use of radioactive isotopes for the elucidation of cellulose reactions. Some applications of the tracer technique were mentioned earlier (*101*, *143*). In addition, Stamm and Borgin (*164*) by using radioactive zinc (Zn^{65}), were able to demonstrate that cellulose takes up the sodium zincate complex from its aqueous solution by way of physical adsorption and not by chemical combination. On the program of the International Congress was a paper by Greathouse, Harris, and coworkers on the preparation of radioactive cellulose by allowing bacteria, such as *Acetobacter xylinum*, to grow on radioactive sugars or alcohols, such as mannitol. Undoubtedly, such labeled cellulose can serve as a useful starting material for further studies of cellulose reactions [cf. (*165*)].

Tracer technique is also being developed for the study of biochemical reactions and for throwing much needed light upon the synthesis of cellulose in the living plant and in animal organisms.

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Some Observations on the Kinetic Coefficient of Friction of Paper

GEOFFREY BROUGHTON and JOAN L. GREGG

Little systematic work on the kinetic coefficient of friction of paper appears to have been done. In the present work, the friction meter developed by E. C. Dreby has been adapted to paper. The coefficient of friction was found to increase with increasing relative humidity, beating, rosin content, and filler content. It decreased on medium calendering with addition of fatty acids and paraffin. Different furnishes showed friction coefficients varying considerably. The cross direction also showed a higher coefficient than did the machine direction. Gloss and surface roughness measurements were also made upon some of the papers studied.

A STUDY of the more recent literature, with the exception of one paper (*1*), failed to reveal any information upon the coefficient of friction of paper. Nevertheless, the kinetic coefficient of friction must be of considerable importance in calendering, supercalendering, printing, and other operations where the surface of a paper sheet moves against a similar sheet or a foreign surface. It must also be a factor in erasability. For these reasons a preliminary study has been made of the kinetic coefficients of friction of a number of papers, with and without various additives.

All surfaces are rough; the asperities being submicroscopic, 10^{-5} cm. or less, for highly finished surfaces to macroscopic, 10^{-2} cm. or more, for the rougher surfaces. It is generally agreed that these hills and valleys play a major role in kinetic friction. This phenomenon has been investigated extensively for metals by Bowden and his collaborators (*2*). Two types of external friction are differentiated, static and kinetic. The former is the force that must be overcome before a body can be moved and is usually measured on an inclined plane. Kinetic friction is the force required to maintain motion between two surfaces and is independent of the area of contact between the two solid bodies involved. It is, however, proportional to the load between the surfaces or force = μ (load on surface), where μ is a constant, the coefficient of friction; and in the case of surfaces in motion, the coefficient of kinetic friction.

It can be shown (*3*) that $\mu = h/\lambda$, where h is the average height of the asperities in the surface, and λ is the average distance between them. It would be expected, therefore, that the coefficient of friction would bear some relationship to such properties as gloss, which are also functions of surface smoothness. Delcroix (*1*) has made some generalizations along these lines, asserting that the fibers themselves have little influence upon the coefficient; fillers affect the coefficient according to their particle shape, sharp-edged particles such as calcium sulphate increasing it, lamellar particles, e.g., talc, decreasing it, and round particles, as an example of which he cites kaoline, having little effect upon the kinetic coefficient although paraffin markedly lowered it. Some of these findings appear to be the reverse of theoretical expectations.

APPARATUS

The machine originally devised by Dreby (*4*) for work on textiles was found to be readily adaptable for use with paper. Figure 1 shows the apparatus, one strip of paper, 3 by 9 in., being clamped to a torsion bar a, while another strip of the same size held in the drum clamp b, can be pulled steadily under the first strip by rotating the handle c. A 1-lb. weight d rests on the top sheet of paper. The torsion bar records the pull of the upper sheet and can be calibrated by means of a pulley and weights. For the present apparatus, if I is the deflection indicated by the gage e in thousandths of an inch, F is the force required to maintain motion in ounces, μ is the coefficient of friction, and P the load in ounces.

$$F = 2I/4$$
$$\mu = I/4P$$
$$\mu = I/4 \times 16$$
$$\mu = I/64$$

In other words, the steady deflection obtained when the

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crank was turned, divided by 64, gave directly the coefficient of friction.

MATERIALS AND METHOD

A number of handsheets made up without addition of any chemicals were used to compare the frictional coefficients of different fibers. Handsheets were also used to investigate the effect of rosin and filler on friction. Most handsheets had a caliper of 8 to 9 thousandths and a basis weight of about 28 lb. per 1000 sq. ft.

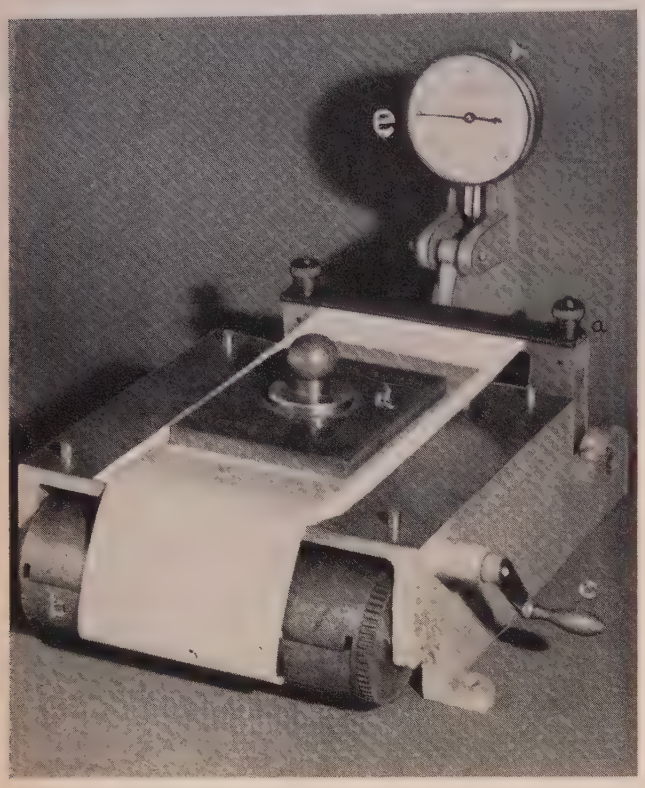

Fig. 1.

Some typical purchased commercial papers were also used to check the influence of machine direction and felt and wire sides.

After some preliminary work, most friction coefficients were determined at 65% relative humidity and 70°F. using the paper under investigation for the top surface and a copper bedplate for the bottom surface. Five passes were made on each of five samples and the average of the readings taken. Questionable values were rejected by the method of Dean and Dixon (*5*), designed particularly for use with a small number of observations. Using this method, out of 3035 values, 81 were discarded; i.e., less than 2.7%.

Readings were made on some of the calendered papers using the Profilometer, which, by means of a diamond tracing point riding lightly on the paper surface, gives a measure of average surface roughness in microinches. Gloss readings were also taken on some of the papers studied according to the TAPPI standard.

RESULTS

Successive Runs in the Friction Meter

In a first series of experiments the same sheet of paper was given many successive runs on the friction meter. The paper was used for the top surface, felt side down, and the copper bedplate for the bottom surface. The paper attached to the crank was felt side up, and the direction of pull was in the machine direction. To denote this, the abbreviation M.F.-F. will be used, indicating pull in the machine direction, felt side to felt side.

Figure 2 shows the results obtained on a bond (25% rag), and 25% rag writing, and a groundwood sheet, all machine made, and on a soda pulp handsheet containing no chemicals. The log-log plot seems to indicate a tendency to step-wise decrease in coefficient of friction, irrespective of type of paper. Furthermore, there is no indication that an asymptotic value is approached although the initial decrease is most marked.

Because of this behavior, it was decided for later determinations to make five series of five successive runs. The average of the first readings of these five series is the figure given in subsequent tables and figures unless otherwise noted.

Effect of Relative Humidity

It was expected that high relative humidity, causing fiber swelling, would probably result in higher coefficients of friction and such was found to be the case (Fig. 3). The bond paper, which had the highest coefficient of friction at 65% R. H., had the lowest at 20 and 90% R. H's. Runs were made M.F.-F. in a relative humidity cabinet after an 8-hr. conditioning.

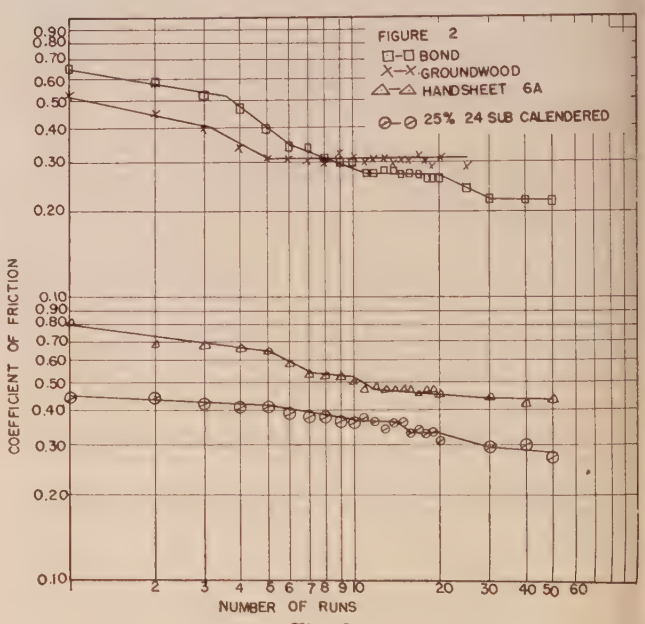

Fig. 2.

All subsequent experiments were made at 65% R. H. and 70°F.

Frictional Coefficients Against Other Materials

The coefficients of friction of a bond paper (25% rag) were determined for paper against paper; i.e., paper above and below the sheet being pulled by the drum, paper against lucite, by placing lucite plates above and below the moving sheet, and paper against

copper, by placing a copper plate above the moving sheet (the bedplate is normally copper). As stated earlier, most runs were made with the moving sheet between an upper sheet of the same paper held by the torsion bar and the copper bedplate underneath.

The results of these experiments are shown in Table I. Paper versus paper on both sides gives the highest

Table I. Effect of Material on Coefficient of Friction

Surface	Run	Series 1	2	3	4	5	Average
Bond paper vs. bond paper, M.F-F.	1	0.90	1.14	0.84	1.02	1.25	1.03
	2	0.86	0.93	0.76	0.86	0.98	0.88
	3	0.76	0.78	0.76	..	0.78	0.76
	4	0.74	0.76	0.70	0.82	0.75	0.75
	5	0.69	0.67	0.67	..	0.69	0.68
Bond paper vs. copper, both sides M.	1	0.40	0.38	0.36	0.32	0.32	0.36
	2	0.42	0.37	0.37	0.33	0.33	0.36
	3	0.38	0.38	0.37	0.34	0.33	0.36
	4	0.38	0.37	0.35	0.34	0.32	0.33
	5	0.39	0.36	0.36	0.36	0.34	0.36
Bond paper vs. lucite, both sides M.	1	0.23	0.22	0.23	0.23	0.24	0.23
	2	0.24	0.23	0.24	0.21	0.24	0.23
	3	0.23	0.24	0.23	0.22	0.24	0.23
	4	0.23	0.24	0.24	0.23	0.23	0.23
	5	0.25	0.24	0.24	0.23	0.24	0.24
Bond paper vs. bond paper and copper, M.F-F.	1	0.56	0.56	0.55	0.55	0.56	0.56
	2	0.52	0.52	0.54	0.50	0.52	0.52
	3	0.51	0.49	0.49	0.50	0.50	0.50
	4	0.50	0.46	0.45	0.46	0.47	0.47
	5	0.47	0.45	0.45	0.44	0.47	0.46

coefficients and also the least reproducible; the coefficient also shows the greatest drop on repetitive runs. When a smooth polished copper plate is substituted for paper on one side, the coefficient falls and becomes more reproducible, and this trend is carried further when paper is replaced on both sides by copper or lucite.

It was thought that the purposes of this investigation would be best served by determining the friction using paper on one side and copper on the other, and subsequent experiments were run in this manner.

Machine Direction and Surface

Table II shows average initial coefficients of friction for six types of paper in common use; machine direction, cross direction, wire to wire, wire to felt, and felt to felt. In general, the lightly sized sheets,

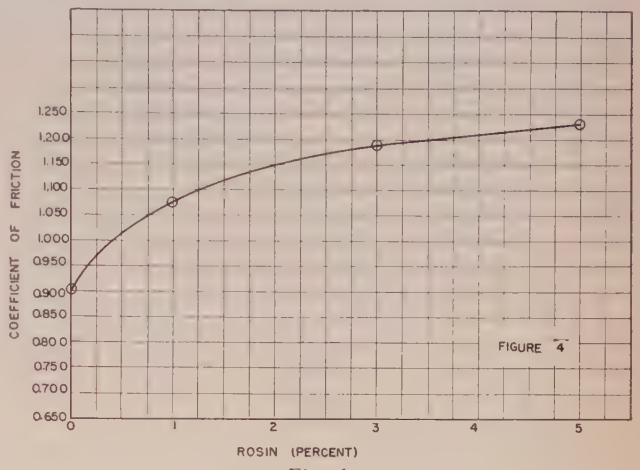

Fig. 4.

Table II. Effect of Machine Direction, Wire and Felt Sides on Coefficient of Friction

Paper	Basis weight, lb./1000 sq. ft.	Caliper, in.	Machine direction F.-F.	F.-W.	W.-W.	Cross direction F.-F.	F.-W.	W.-W.
Bond	16.2	0.0039	0.67	0.60	0.62	0.63	0.61	0.57
Groundwood	10.6	0.0034	0.50	0.56	0.54	0.56	0.55	0.57
Onionskin	6.9	0.0018	0.53	0.51	0.52	0.54	0.56	0.60
Kraft wrapping	13.6	0.0043	0.52	0.53	0.56	0.54	0.60	0.60
Blotting	60.7	0.0212	0.58	0.62	0.63	0.63	0.62	0.61
Coated book	21.2	0.0034	0.45	0.45	0.46	0.51	0.50	0.50

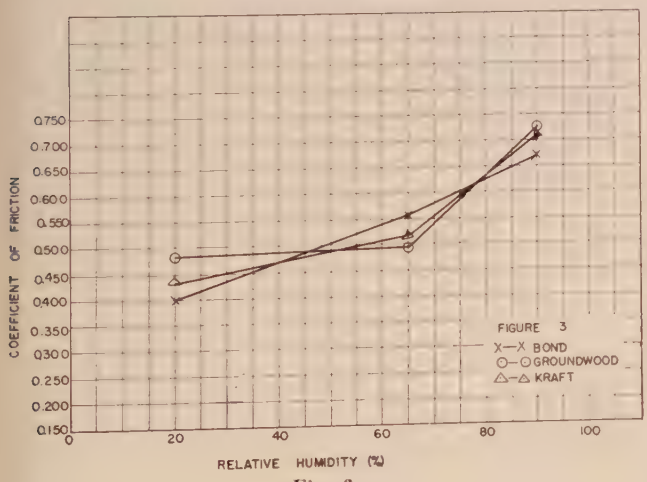

Fig. 3.

such as the groundwood, kraft, and blotting, show a higher coefficient of friction on the W.-W. than on the F.-F. runs. The exception is blotting in the cross direction. These sheets also show a consistently higher friction in the cross than in the machine direction both for W.-W. and F.-F. This last observation is also true for onionskin, and the coefficient of friction is higher in the W.-W. than in the F.-F. for the cross direction. The coated paper, as might be expected, does not show as much variation between the wire and felt sides as do most of the uncoated papers; it does, however, show a difference between machine and cross directions. No difference was observed between coefficients of friction when the paper was reversed through 180° in the machine direction; i.e., parallel and antiparallel to the machine direction.

Since in most of the cases studied the cross-direction friction was higher than the machine-direction and the F.-F. was different from the W.-W., it was decided to standardize upon the machine-direction F.-F. arrangement in subsequent experiments.

Effect of Pulp. In order to trace the influence of different fibers, if any, a series of handsheets were made

up to a basis weight of 28 lb. per 1000 sq. ft. without the addition of size of chemical. Results of friction measurements are shown in Table III; while fiber undoubtedly influences the coefficient, it is hard to draw any conclusions.

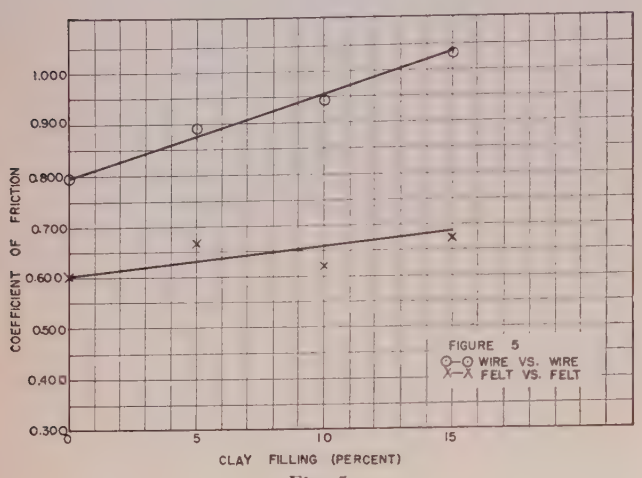

Fig. 5.

Effect of Beating. Four of the pulps in Table III were beaten, handsheets again made, and the coefficients of friction determined (Table IV). Cross comparison shows that the friction showed a considerable change only in the case of esparto. Only in the case of northern kraft did the coefficient show a decrease.

Table III. Effect of Fiber on Coefficient of Friction

Pulp	*Basis weight, lb./1000 sq. ft.*	*Caliper, 1/1000 in.*	*Friction coefficient*
Sulphite (West Coast)	27.5	8.9	0.680
Northern kraft A	28.0	8.8	0.875
Northern kraft B	27.4	8.3	0.760
Soda	27.8	9.8	0.830
Esparto	28.0	11.9	0.645
Rag stock	27.7	8.5	0.755

Effect of Rosin Size. A series of sulphite handsheets with varying rosin contents gave the results shown in Fig. 4, coefficient of friction rising steadily with rosin addition to the furnish.

Table IV. Effect of Beating on Coefficient of Friction

Pulp	*Basis weight, lb./1000 sq. ft.*	*Caliper, 1/1000 in.*	*Williams freeness, sec.*	*Friction coefficient*
Sulphite (West Coast)	28.4	8.7	106.6	0.690
Northern kraft A	28.1	8.2	97.7	0.820
Soda	28.5	8.8	47.4	0.870
Esparto	28.7	9.8	106.1	0.745

Effect of Clay Loading. A series of sulphite handsheets were also made up with varying additions of clay to the beater. Results are shown in Fig. 5. As would be expected, the coefficient of friction is decidedly greater on the wire side and rises steadily with increasing clay content. Apparently, clay can alter significantly the frictional properties of the sheet.

Supercalendering. Through the courtesy of the Byron Weston Co., a number of samples of their uncalendered paper were obtained. The coefficient of friction and gloss were measured and the sheets then passed through a large supercalender equipped with steel and cotton rolls (Table V). Pressure, unfortunately, could not be measured.

Since these preliminary experiments showed a consistent decrease in frictional coefficient with calendering, some more carefully controlled experiments were run on a B. F. Perkins laboratory supercalender. The samples were run in the machine direction wire side down on an 11-in. steel roll, the felt side facing a 12-in. cotton roll. Three nip pressures with three, six, and nine passes in each case were run.

Table V

Paper	*Fiber content, % rag*	*Basis weight, lb./1000 sq. ft.*	*Caliper, 1/1000 in.*	*Before calendering* *Friction coefficient*	*Before calendering* *Gloss*	*After calendering* *Friction coefficient*	*After calendering* *Gloss*
Parchment	100	36.7	8.2	0.58	6.0	0.57	11.0
Ledger	100	29.0	5.7	0.61	5.5	0.59	14.0
Index	50	62.3	13.3	0.63	5.0	0.58	12.0
24 Substance	50	18.2	4.3	0.60	7.0	0.57	10.0
24 Substance	25	16.6	4.1	0.57	7.0	0.54	10.5

Friction coefficients were determined M.W.-W., since it was desired to follow friction on the roughest surface. The Physicists Research Co. were kind enough to make measurements of roughness height (root means square) on the wire side of a number of the samples. A Profilometer was used with a Q Amplimeter, V Motortrace, and LA Tracer.

Results are plotted in Fig. 6, the paper used being a specialty kraft, 29.6 lb. per 1000 sq. ft. Changes in gloss and caliper are plotted in Fig. 7.

The first calendering that the paper receives lowers the coefficient of friction the greatest amount. In general, the surface roughness follows the friction coefficient, an indication of the basic correctness of the belief that friction is caused by the surface asperities

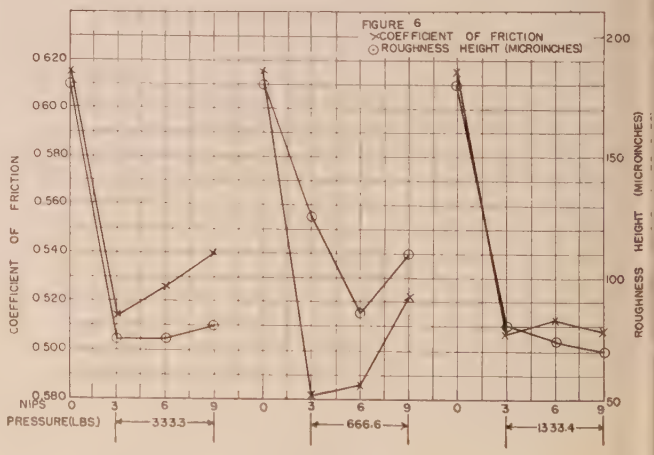

Fig. 6.

of the sheet. When the sheet is given more calendering, six or nine nips, without additional increase in pressure, the coefficient of friction increases although the gloss continues to rise. At the highest pressure, 1333.4 lb. per nip in. coefficient of friction does not tend to rise with more passes as at lower pressures.

Table VI. Effect of Supercalendering on Some Kraft Specialty Papers

Treatment	Paper 1		Paper 2		Paper 3		Paper 4		Paper 5	
	Friction coefficient	Gloss	Friction coefficient	Gloss	Friction coefficient	Gloss	Friction coefficient	Gloss	Friction coefficient	Gloss
Uncalendered	0.57	7.5	0.58	8.5	0.61	8.5	0 53	8.0	0.56	8.5
333 lb./in. 3 nips	0.43	18.0	0.47	16.0	0.51	22.0	0.47	20.0	0.45	16.0
333 lb./in. 6 nips	0.47	20.0	0.50	22.0	0.53	25.5	0.50	20.5	0.52	17.0
333 lb./in. 9 nips	0.47	21.0	0.51	25.0	0.54	25.0	0.51	19.5	0.51	18.0
667 lb./in. 3 nips	0.44	20.5	0.48	22.0	0.48	15.0	0.52	22.0	0.48	23.5
667 lb./in. 6 nips	0.46	22.5	0.50	15.5	0.49	15.0	0.48	20.0	0.48	16.5
667 lb./in. 9 nips	0.47	22.5	0.49	16.5	0.52	18.0	0.50	20.5	0.50	17.0
1333 lb./in. 3 nips	0.47	21.5	0.52	27.5	0.51	27.0	0.50	19.0	0.52	26.5
1333 lb./in. 6 nips	0.47	26.5	0.51	28.5	0.51	28.5	0.49	24.5	0.52	29.5
1333 lb./in. 9 nips	0.48	26.5	0.47	28.0	0.51	28.0	0.46	26.0	0.51	30.0

This was confirmed by more measurements on some further papers (Table VI), kindly made available by the St. Regis Paper Co. (Nashua River Div.). Here it is seen that at the highest nip pressure, the coefficient of friction remains constant or tends to decrease with more nip passes. It is also interesting to note that all the papers seem to be tending to a common coefficient of friction and gloss value in spite of initial calipers varying from 4.7 to 10.2 thousandths and basis weights from 14.9 to 29.6 lb. per thousand sq. ft.

Table VII. Effect of Coating and Calendering

	Basis weight, lb./1000 sq. ft.	Caliper, 1/100 in.	Friction coefficient	Surface roughness, microin.
Uncoated	27.2	5.4	0.27	70
Coated	..	7.6	0.66	12
Supercalendered	..	5.9	0.56	9.5

It may be that while the first imposition of pressure causes mainly rearrangement of the surface fibers, continued imposition or higher pressures cause more deep seated rearrangement which may even lead to an increase of surface roughness.

Coating and Coefficient of Friction. Coating can definitely change the coefficient of friction. Table VII shows results on a specialty paper, before and after coating and after supercalendering.

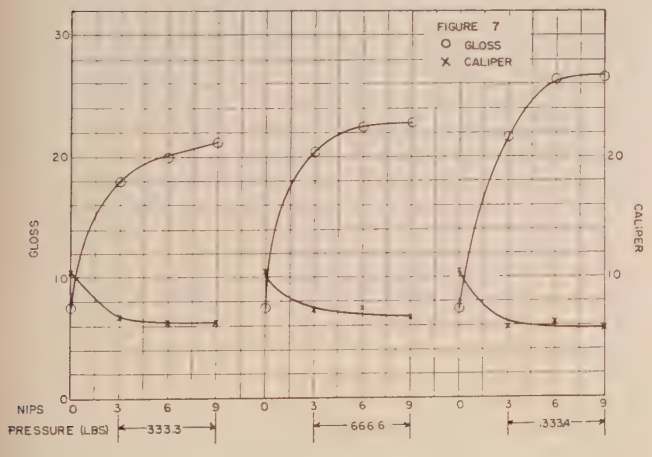

Fig. 7.

Friction Reduction. Modifying agents are sometimes added to coating mixes to give better supercalendering, and it was thought to be of some interest to investigate whether or not the coefficient of friction could be changed readily by addition of simple compounds.

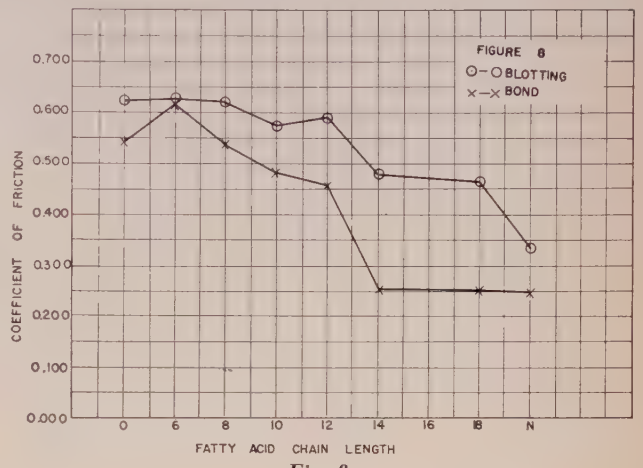

Fig. 8.

Previous work on film friction has shown the fatty acids to be of particular interest, the coefficient of friction of metals decreasing as the hydrocarbon chain of the fatty acid used as a lubricant increases. For this reason, solutions of the fatty acids and of paraffin wax (which may be regarded as a fatty acid with an infinitely long hydrocarbon chain) were made up, sheets of bond and blotting paper dipped in them for 5 min., dried, conditioned, and run on the friction meter. Figure 8 shows similar results to those obtained on metals. The lower acids affect the friction only in a minor way, the big decrease coming with the change from lauric to myristic. Paraffin wax, as to be expected, shows the lowest coefficient of friction.

To summarize, it appears that the Dreby friction meter can be applied to paper without major changes. Papers vary in coefficient of friction widely, two and one-half fold variation having been observed on commercial papers in the present study. Some of the factors, e.g., furnish, size, filler, relative humidity, calendering, coating, additives, influencing friction, have been pointed out and investigated, and the importance of machine direction and paper two-sidedness noted. It is believed that further work may show the importance of frictional phenomena in the behavior of paper in printing, in the finishing room, and other places where there is motion of a paper web or sheet.

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Digester Corrosion Measurement

NICHOLAS SHOUMATOFF

Three semiannual sets of thickness measurements of a typical sulphate digester are presented and analyzed in detail by statistical methods. It is concluded that the inspection procedure based on such measurements is a powerful and reliable tool for the study of digester corrosion.

THE main purpose of this paper is to present data on sequential wall thickness measurements, with particular emphasis on quantitative analysis of the data, as an illustration of the study of corrosion in a typical sulphate pulp digester. Investigation is made of the validity of the inspection procedure with the Audigage thickness tester, of the local variations of thickness and corrosion within the vessel and of the best means for interpreting routine inspection data to predict the remaining service life of various portions of the vessel. To accomplish these objectives the analysis is probably more extensive than required for routine inspections, but it seems to be justified as part of our still exploratory approach to the important problem of digester corrosion. Although no attempt is made to generalize the observed effects these specific results may be of interest for comparison with previous or future studies.

The no. 4 digester at the Luke, Md., mill of the West Virginia Pulp & Paper Co., which is the subject of this study, was placed in service in Feb., 1951. The mill was converted from a modified soda to a full kraft process by placing in service a spray-type recovery unit with electrostatic precipitator in April, 1951.

Extracts of the vessel manufacturer's design and test data are shown in Tables I and II.

INSPECTION PROCEDURE

The inspection procedure closely followed the recommendations of the TAPPI Data Sheet (*2*). The layout of the grid used in locating the test points is shown in Fig. 1. The readings, taken with an Audigage instrument, as recorded from the three inspections, taken at approximately semiannual intervals, by Louis DelaGrange, are shown in Table III. To obtain a complete rectangular pattern for simplicity of statistical analysis, the readings in row 21, which were not complete due to interference with the strainer, but which did not appear to be in any way abnormal, are not included in the present study. The remaining 528 readings provide an entirely ample base for analysis.

The physical location of these points was accomplished by means of plumb lines hung from eight equally spaced studs welded to the vessel at the top of the straight side. The points were located along these plumb lines and readings taken from a collapsible scaffold which was lowered 2 ft. at a time. In the first two sets of readings the points were located along the plum line by measuring off the intervals from one point to the next. It was felt that this might induce cumulative errors in locating the points. For this reason in the third set of readings an improved method was used. With this method the plumb lines were replaced with chains with $1^3/_4$-in. washers inserted into the chains at 2-ft. intervals. The readings were then taken by inserting the $1^3/_8$-in. diameter crystal of the Audigage instrument into the hole of each washer. It is felt that this method provides a convenient and positive means for accurate location of the test points and in addition it saves time. In all three sets of readings the points in the top head were located by measuring upward from the reference studs with a tape measure.

Figure 1 also shows the different types of surfaces observed inside the digester. It is felt that this method of showing the visual inspection results on the same grid as is used for locating the points has an advantage over the method shown in the TAPPI Data Sheet in that it facilitates precise correlation of the visual results with the thickness readings. The possibility of doing this readily is felt to be an important advantage of those instruments which take readings from the inside of the

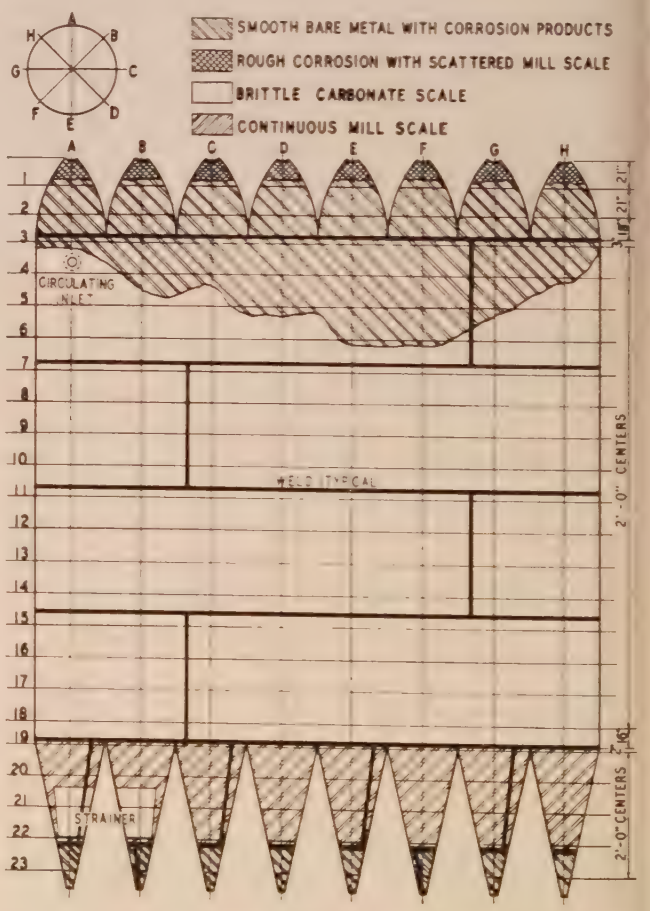

Fig. 1. Test point locations and visual inspection results (Feb., 1952)

NICHOLAS SHOUMATOFF, West Virginia Pulp & Paper Co., New York, N. Y.

Table I. Design Data

1. Inside diameter shell, 11 ft. 3 in.
2. Over-all height, 42 ft. 9 in.
3. Design pressure, 150 p.s.i. plus hydrostatic
4. Hydrostatic test pressure, 330 p.s.i.
5. Material shell and heads, ASTM A 285 Gr. "C" FBQ
6. All fittings and internals, carbon steel
7. Shell thickness, $1^{13}/_{32}$ in.
8. Shell construction, 4 courses, 1 plate per course
9. Top head thickness, $1^{9}/_{16}$ in.
10. Top head construction, 1 piece, flanged and dished
11. Bottom cone thickness, $1^{5}/_{8}$ in. (top section, 5 ft. 3 in. high) Bottom cone thickness, 1 in. (bottom section, 2 ft. 1 in. high)
12. Bottom cone construction, 71° 38′ included angle, no knuckle radius
13. Code stamp, API-ASME
14. Factor of safety, 4
15. Joint efficiency, 85%
16. Corrosion allowance (shell), $^{7}/_{16}$ in.
17. Vessel stress relieved but not x-rayed

vessel as compared with those which are used from the outside.

The number of significant figures in the readings as recorded in Table III is limited only by the accuracy in reading a slide rule in connection with the use of the instrument and has no relation to the precision of the readings as compared with the actual thicknesses. Since statistical errors are not necessarily related to each other in exact multiples of ten, any arbitrary reduction of the number of significant figures in the raw data would artificially change the statistical content of these data, which it is the purpose of this study to investigate. Therefore no such arbitrary change in the original records is proposed.

ANALYTIC APPROACH

Inspection of Table III reveals the presence of obviously statistical errors in the thickness readings. These errors are greatly magnified in relation to corrosion rates based on small percentage changes in thickness values. It is evidently necessary to investigate the quantitative statistical character of the accumulated data before the evidence of these readings can be intelligently interpreted.

The method of investigation used, which is called analysis of variance and covariance (*1*), involves only five basic algebraic relationships. The first step is to classify the readings into groups according to all available known causes of variation and to calculate the amount of variation in the data according to each of these causes. The following symbols are used:

x = time in years
y = thickness in inches
$\bar{y}$ = average thickness of a group
$\bar{\bar{y}}$ = average thickness all readings
N = total number of readings
m = number of readings in a group
S = summation

The contribution of each group to the amount of variation due to an assignable cause is as follows:

$$m(\bar{y} - \bar{\bar{y}})^2 = \frac{(Sy)^2}{m} - m\bar{\bar{y}}^2 \qquad (1)$$

This equation is applied successively to obtain the following results as shown in Table IV:

1. Total variation of thickness among mean values at each point.
2. Vertical variation of thickness equals total variation among mean values of each horizontal row.
3. Horizontal variation of thickness (from mean values of vertical columns).
4. Thickness variation among plates. For this calculation each course in the cone is considered as a single plate, so that the resulting variation is all in the vertical direction.
5. Random local variation of thickness, equals "total" minus the sum of "vertical" and "horizontal."
6. Vertical variation within plates equals "vertical" minus "plates."
7. Total variation due to general corrosion equals variation among the means for the entire vessel of the three successive sets of readings.

Calculation of average corrosion rates is greatly simplified by choosing the time coordinates with respect to the mean time values (see Table III). Thus the best straight line through the thickness values plotted against time, by the method of least squares or linear regression, is

$$Y = (\bar{y} + bx) \qquad (2)$$

where the slope, or linear corrosion rate, is

$$b = \frac{Sxy}{Sx^2} \qquad (3)$$

The values of this slope calculated for each point, row, and column, and for the entire vessel, are shown in Table V. The squared deviations in thickness due to any one of these slopes are

$$S(bx)^2 = \frac{(Sxy)^2}{Sx^2} \qquad (4)$$

With this equation the analysis may be completed to show the effects of variations in corrosion rates in Table IV, as follows:

1. Linear component of general corrosion is calculated from the slope of the regression line for the whole vessel.
2. This quantity subtracted from the total variation due to general corrosion (above) leaves the nonlinear component, that is, the effect of *change* in the corrosion rate.
3. The deviations due to the slopes at the points, rows, and columns, are calculated, and from each of these three quantities the linear component of general corrosion is deducted to obtain the effect of *variation* in corrosion rates vertically, horizontally, and locally, exactly as was done with the thickness variations.
4. The vessel is divided into three major corrosion zones, and the effect of variation in corrosion among these zones is calcu-

Table II. Steel Analyses

	Chemicals, %				Strength, p.s.i.	
	C	*Mn*	*P*	*S*	*Yield*	*Tensile*
Specified (max.)	0.30	0.80	0.035	0.04	...	65,000
Specified (min.)	...	...	...	...	30,000	55,000
Actual:						
Top head	0.14	0.50	0.014	0.032	37,600	60,700
Shell plate I	0.19	0.46	0.011	0.036	37,330	60,440
Shell plate II	0.19	0.46	0.011	0.036	38,530	62,900
Shell plate III	0.19	0.46	0.011	0.036	36,490	59,950
Shell plate IV	0.19	0.46	0.011	0.036	35,940	60,390
Four upper cone plates	0.23	0.50	0.010	0.029	33,340	60,760
Two lower cone plates	0.22	0.48	0.009	0.030	33,720	61,170

lated and then deducted from the total vertical to obtain the effect of vertical variation of corrosion within zones. (See Table VI for nominal horizontal boundaries of zones.)

The degrees of freedom corresponding to each of the above causes of variation are shown in Table IV. In each case this is the number of independent observations or parameters less the number of parameters with respect to which deviations are calculated. The squared deviations are divided by the degrees of freedom to obtain the mean-square deviation, or variance.

The total variation among all the readings is

$$S(y - \bar{y})^2 = Sy^2 - \frac{(Sy)^2}{N} \qquad (5)$$

and its degrees of freedom are one less than the total number of readings. The squared deviations and degrees of freedom for each assignable cause of variation are subtracted from these totals until a minimum residual unassignable variance is obtained which is assumed to represent the experimental error. It is

Table III. Record of Audigage Thickness Measurements in Inches on No. 4 Digester, Luke, Md.

First reading each point taken July 12, 1951, time coordinate, −0.53 years; second reading each point taken Feb. 15, 1952, time coordinate, +0.07 years; third reading each point taken July 7, 1952; time coordinate, +0.46 years

Row	Column A	B	C	D	E	F	G	H
1	1.660	1.660	1.675	1.666	1.666	1.666	1.630	1.666
	1.645	1.645	1.625	1.665	1.625	1.645	1.625	1.665
	1.592	1.650	1.663	1.650	1.650	1.635	1.554	1.580
2	1.630	1.619	1.666	1.630	1.608	1.630	1.630	1.666
	1.645	1.570	1.645	1.590	1.610	1.625	1.645	1.625
	1.518	1.492	1.569	1.503	1.529	1.540	1.554	1.592
3	1.465	1.455	1.455	1.455	1.455	1.444	1.485	1.465
	1.443	1.423	1.423	1.423	1.412	1.391	1.443	1.443
	1.392	1.351	1.351	1.351	1.351	1.331	1.342	1.383
4	1.458	1.458	1.458	1.467	1.458	1.440	1.505	1.495
	1.469	1.434	1.457	1.434	1.412	1.412	1.480	1.491
	1.470	1.413	1.392	1.392	1.372	1.360	1.425	1.503
5	1.476	1.458	1.467	1.458	1.440	1.440	1.495	1.495
	1.469	1.469	1.469	1.434	1.434	1.423	1.469	1.500
	1.458	1.434	1.445	1.413	1.392	1.383	1.400	1.480
6	1.458	1.458	1.448	1.458	1.467	1.448	1.495	1.505
	1.480	1.445	1.458	1.469	1.434	1.412	1.500	1.503
	1.480	1.445	1.434	1.445	1.458	1.400	1.470	1.458
7	1.440	1.448	1.448	1.448	1.440	1.440	1.448	1.431
	1.445	1.434	1.445	1.445	1.443	1.434	1.434	1.445
	1.458	1.413	1.425	1.434	1.425	1.434	1.425	1.434
8	1.476	1.448	1.440	1.448	1.448	1.448	1.467	1.448
	1.503	1.492	1.445	1.458	1.458	1.458	1.458	1.480
	1.434	1.503	1.413	1.445	1.434	1.434	1.480	1.445
9	1.467	1.495	1.458	1.458	1.458	1.458	1.458	1.467
	1.492	1.480	1.434	1.458	1.445	1.458	1.445	1.458
	1.503	1.503	1.434	1.434	1.445	1.434	1.434	1.434
10	1.458	1.476	1.448	1.448	1.448	1.448	1.458	1.458
	1.503	1.475	1.434	1.458	1.445	1.445	1.458	1.458
	1.503	1.425	1.434	1.434	1.425	1.445	1.425	1.470
11	1.405	1.405	1.405	1.422	1.440	1.440	1.388	1.388
	1.413	1.413	1.413	1.445	1.434	1.470	1.392	1.392
	1.400	1.392	1.383	1.440	1.445	1.413	1.383	1.392
12	1.413	1.413	1.405	1.422	1.422	1.440	1.405	1.396
	1.413	1.392	1.392	1.400	1.400	1.480	1.400	1.400
	1.392	1.392	1.392	1.392	1.492	1.470	1.392	1.400
13	1.413	1.413	1.405	1.422	1.422	1.405	1.405	1.405
	1.413	1.425	1.372	1.400	1.434	1.480	1.413	1.413
	1.392	1.400	1.392	1.503	1.434	1.480	1.400	1.392
14	1.413	1.422	1.413	1.422	1.458	1.405	1.413	1.413
	1.425	1.396	1.445	1.400	1.458	1.480	1.413	1.425
	1.413	1.392	1.434	1.400	1.425	1.400	1.392	1.400
15	1.405	1.431	1.422	1.422	1.422	1.440	1.422	1.413
	1.425	1.434	1.413	1.413	1.434	1.413	1.425	1.413
	1.434	1.383	1.413	1.425	1.413	1.413	1.400	1.413
16	1.467	1.448	1.431	1.440	1.440	1.422	1.442	1.440
	1.413	1.458	1.400	1.434	1.445	1.434	1.434	1.425
	1.434	1.445	1.425	1.425	1.425	1.413	1.425	1.413
17	1.458	1.448	1.440	1.448	1.431	1.440	1.422	1.440
	1.492	1.480	1.413	1.425	1.434	1.413	1.445	1.434
	1.470	1.518	1.434	1.434	1.434	1.445	1.445	1.425
18	1.458	1.440	1.431	1.448	1.431	1.431	1.440	1.440
	1.480	1.425	1.413	1.425	1.434	1.413	1.425	1.445
	1.470	1.434	1.425	1.425	1.425	1.434	1.445	1.458
19	1.666	1.727	1.654	1.666	1.666	1.666	1.666	1.666
	1.680	1.650	1.680	1.663	1.650	1.650	1.663	1.663
	1.663	1.663	1.635	1.650	1.635	1.680	1.663	1.663
20	1.666	1.666	1.666	1.666	1.666	1.666	1.666	1.666
	1.635	1.680	1.663	1.663	1.656	1.663	1.663	1.680
	1.635	1.650	1.635	1.650	1.663	1.680	1.680	1.650
22	1.642	1.654	1.642	1.666	1.666	1.642	1.666	1.642
	1.620	1.635	1.650	1.680	1.650	1.663	1.663	1.614
	1.663	1.608	1.635	1.650	1.680	1.663	1.663	1.650
23	1.619	1.597	1.597	1.587	1.597	1.597	1.597	1.576
	1.540	1.580	1.592	1.569	1.608	1.608	1.580	1.554
	1.554	1.554	1.540	1.580	1.569	1.554	1.540	1.569

Table IV. Analysis of Audigage Thickness Measurements on No. 4 Digester, Luke Md., Based on Readings July, 1951, Feb., 1952, July, 1952

	Causes of variation in thickness readings	*Squared deviations*	*Degrees freedom*	*Variance*	*Variance ratio*		
					Observed	*95% limit*	*Significance*
1.	Thickness variation among plates	4.232601	6	0.706	2220	2.11	Yes
2.	Vertical variation of thickness within plates	0.070166	15	0.00468	14.7	1.70	Yes
3.	Horizontal variation of thickness	0.007818	7	0.00112	3.52	2.04	Yes
4.	Random local variation of thickness	0.142061	147	0.000966	3.04	1.27	Yes
5.	Linear component of general corrosion	0.037852	1	0.0378	119	3.87	Yes
6.	Nonlinear component of general corrosion	0.006932	1	0.00693	21.8	3.87	Yes
7.	Variation of linear corrosion among corrosion zones	0.052243	2	0.0261	82.1	3.03	Yes
8.	Vertical variation of linear corrosion within zones	0.018914	19	0.000997	3.14	1.63	Yes
9.	Horizontal variation of linear corrosion	0.002472	7	0.000353	1.11	2.04	No
10.	Experimental error—by difference	0.102082	322	0.000318	...	...	...
11.	Total variation among all readings	4.673141	527	0.00888	...	...	...
12.	Random local variation of linear corrosion	0.044289	147	0.000301	0.947	0.79	No

this maximum degree of precision obtainable from the particular set of data whose testimony must be considered in arriving at conclusions. Thus the effect of random local variation in corrosion was not deducted to arrive at the experimental error in Table IV because its variance is less than that of error. Each variance is then tested for significance by expressing it as a ratio of the variance due to error and comparing this ratio with random sampling limits at the conventional 95% confidence level, obtained from published tables (*1*).

The power of the testimony of the three sets of readings can be appreciated when it is considered that, after elimination of the variations due to assignable causes, there remains the equivalent of 322 independent measurements at the same time and place as a basis for calculating the experimental precision. Thus, in calculating the limits of error for thicknesses and corrosion rates, small sampling functions may be neglected and the limits of error at 95% confidence may be taken as simply twice the standard deviation s (square root of the variance). Therefore, the error for a single reading is

$$2s = 2(0.000318)^{1/2} = 0.0357 \text{ in.}$$

which is within the 3% accuracy stated by the instrument manufacturer. For the error of the regression slope at one point,

$$2s_b = \frac{2s}{(Sx^2)^{1/2}} = \frac{0.0357}{(0.4974)^{1/2}} = 0.0506 \text{ i.p.y.}$$

For the difference between two readings, the error is

$$2s_d = 2s(1.414) = 0.0505 \text{ in.}$$

For average values involving more than one point, or additional readings which may be taken to recheck the same point or explore its immediate vicinity, the above limits are considerably reduced varying inversely as the square root of the number of readings.

The error limits for the regression slope and for the difference of two readings are virtually identical because, with the three approximately semiannual readings, the middle one contributed very little to the determination of the slope. On the other hand, while this slope is expressed directly in inches per year, the difference of two readings must be divided by the time interval to obtain a corrosion rate This is the most obvious reason why the error limit of corrosion rate is greater with two semiannual readings than with three. On the other hand, the above error limits for the difference of two readings are based on the evidence of all three complete sets of readings, and there would be an additional loss of precision if only two sets of readings were available for calculating the estimate of error, due to reduction in the residual degrees of freedom.

Table V. Linear Corrosion Rates in Inches per Year on No. 4 Digester, Luke, Md., Based on Readings July, 1951, Feb., 1952, July, 1952

Negative sign means loss of thickness

Row	*Column*								
	A	*B*	*C*	*D*	*E*	*F*	*G*	*H*	*Average*
1	−0.0650	−0.0134	−0.0181	−0.0149	−0.0206	−0.0316	−0.0710	−0.0797	−0.0390
2	−0.1015	−0.1243	−0.0927	−0.1231	−0.0728	−0.0839	−0.0682	−0.0742	−0.0926
3	−0.0706	−0.1007	−0.1007	−0.1022	−0.1120	−0.1382	−0.07893	−0.1005	
4	+0.0126	−0.0449	−0.0612	−0.0740	−0.0860	−0.0779	−0.0775	+0.0068	−0.0503
5	−0.0176	−0.0206	−0.0201	−0.0449	−0.0452	−0.0551	−0.0915	−0.0132	−0.0385
6	+0.0234	−0.0139	−0.0115	−0.0105	−0.0130	−0.0495	−0.0224	−0.0437	−0.0176
7	+0.0174	−0.0343	−0.0217	−0.0134	−0.0134	−0.0064	−0.0232	+0.0047	−0.0113
8	−0.0350	+0.0571	−0.0243	−0.0014	−0.0115	−0.0115	+0.0108	+0.0017	−0.0018
9	+0.0368	+0.0053	−0.0256	−0.0222	−0.0139	−0.0222	−0.0240	−0.0318	−0.0122
10	+0.0479	−0.0473	−0.0149	−0.0115	−0.0217	−0.0032	−0.0305	+0.0111	−0.0088
11	−0.0035	−0.0109	−0.0192	+0.0199	+0.0038	−0.0207	−0.0041	+0.0043	−0.0038
12	−0.0194	−0.0224	−0.0139	−0.0308	+0.0616	+0.0334	−0.0127	+0.0043	0.0000
13	−0.0194	−0.0103	−0.0167	+0.0718	+0.0128	+0.0799	−0.0035	−0.0109	+0.0130
14	+0.0017	−0.0314	+0.0239	−0.0234	−0.0315	+0.0059	−0.0194	−0.0103	−0.0104
15	+0.0296	−0.0440	−0.0096	+0.0015	−0.0066	−0.0288	−0.0199	0.0000	−0.0097
16	−0.0381	−0.0014	−0.0099	−0.0147	−0.0132	−0.0066	−0.0168	−0.0271	−0.0160
17	+0.0016	+0.0692	−0.0092	−0.0162	+0.0032	+0.0008	+0.0245	−0.0147	+0.0092
18	+0.0014	−0.0077	−0.0081	−0.0245	−0.0051	+0.0002	+0.0025	−0.0174	−0.0014
19	−0.0008	−0.0700	−0.0139	−0.0152	−0.0309	+0.0107	−0.0032	−0.0032	−0.0158
20	−0.0330	−0.0128	−0.0291	−0.0152	−0.0042	+0.0125	+0.0125	−0.0128	−0.0103
22	+0.0163	−0.0452	−0.0053	−0.0128	+0.0107	+0.0224	−0.0032	+0.0035	−0.0017
23	−0.0712	−0.0422	−0.0534	−0.0090	−0.0243	−0.0382	−0.0551	−0.0096	−0.0379
Average	−0.0118	−0.0256	−0.0252	−0.0221	−0.0192	−0.0174	−0.0288	−0.0162	−0.0208

Table VI. Summary of Thicknesses, Corrosion Rates, and Remaining Life on No. 4 Digester, Lake, Md.

Location	Allowable thickness (1)	Actual thickness July 1951	Actual thickness July 1952	Average corrosion allowance	Linear corrosion rate (3) Feb. to July 1952	Linear corrosion rate (3) Feb. to July 1952	Error	Estimated remaining service life, yr Min.	Estimated remaining service life, yr Max.
Conical nozzle									
Point 2A	1.063	0.850	1.649	1.356	0.0342	0.0179	0.0456	2.4	3.0
Point 3A	1.106	0.516	1.441	1.331	0.0347	0.0179	0.0456	3.3	4.7
Cylindrical zone									
Vapor zone (1-5)									
Row 1	1.063	0.497	1.649	1.322	0.0342	0.0179	0.0456	10.1	21.1
Row 2	1.063	0.850	1.649	1.387	0.0326	0.0179	0.0456	2.6	3.3
Row 3	1.106	0.516	1.469	1.357	0.0311	0.0179	0.0456	3.8	4.8
Row 4	1.106	0.520	1.467	1.419	0.0296	0.0179	0.0456	6.8	10.6
Row 5	1.106	0.525	1.466	1.426	0.0285	0.0179	0.0456	7.0	11.0
Scale zone (6-22)			1.480	1.473	0.0032	0.0044	0.0114		
Cone bottom (23)	1.000	0.236	1.596	1.558	1.322	0.0379	0.0538	13.1	24.6
Whole vessel			1.198	1.477	0.0208	0.0410	0.0097		
Plates:									
Top head	1.563	0.850	1.638	1.631	1.579	0.0658	0.0127	0.1333	0.0322	4.4	5.5	7.3
Shell plate 1	1.106	0.531	1.665	1.449	1.412	0.0517	0.0089	0.0949	0.0228	7.4	9.3	12.3
Shell plate 2	1.106	0.551	1.454	1.457	1.444	0.0893	0.0085	0.0333	0.0228	15.8	26.8	85.6
Shell plate 3	1.106	0.568	1.414	1.420	1.413	0.845	0.0063	0.0089	0.0179	20.6	47.2	...
Shell plate 4	1.106	0.584	1.436	1.432	1.432	0.848	0.0045	0.0089	0.0000	0.0228	36.9	...	...
Top cone plate	1.625	0.720	1.661	1.657	1.651	0.0073	0.934	0.0093	0.0103	0.0077	0.0263	27.3	21.3	...

Notes: (1) Minimum allowable thickness based on 116 p.s.i.g. pressure at steam nozzles. (2) Estimated remaining service life based on corrosion rates Feb. to July, 1952. (3) Limits of error are based on 95% confidence.

DISCUSSION OF RESULTS

Table VI shows a summary of thicknesses, corrosion rates, and estimates of remaining life, together with the limits of error in each case, all derived from the preceding analysis. The estimates of remaining life were calculated by the TAPPI formula (*2*).

The results indicate that the corrosion is localized mainly to the vapor zone with a lesser amount also at the very bottom of the cone. The most striking feature, however, is the pattern of corrosion within the vapor zone as shown by the comparison of the results listed separately for the first five elevations. It is evident that the maximum corrosion is taking place at rows 2 and 3 which are immediately above and below the knuckle radius at the outside diameter of the top head. The corrosion rate falls off markedly both above and below these two critical elevations. This indicates that the corrosion in the vapor zone is not a uniform type of general chemical attack, but must be due to a large extent to some form of mechanical action, such as, washing of the surface, combined with the chemical action of the gases.

A second very significant result of the analysis is that the general corrosion rate has greatly increased during the last 5-month period. The same effect is indicated on Table IV in a more generalized way, where it is seen that the nonlinear component of general corrosion is statistically significant. This means that the corrosion rates calculated by fitting a straight line to the three readings at each point are not acceptable. The best available estimates for the corrosion rates at the present time must, therefore, be based on the comparison of the last two sets of readings only. No explanation was found for the increase of corrosion during the second inspection period. The only known major change in the process during this period is that the external liquor circulation was discontinued. Inspection of the scale pattern in Fig. 1 would indicate that circulation accelerates, rather than retards, corrosion.

On the other hand, the straight line corrosion rates calculated for all three sets of readings provide a more useful index of the corrosion behavior of the vessel during the entire period. It is shown in Table IV that the variation of the corrosion rates between different elevations in the digester is highly significant, even within the same corrosion zone, while on the other hand, the horizontal and random local variations of the corrosion rates are not significant. This means that the best estimate of the corrosion rate at any given point in this vessel is the average of all the rates at the same elevation.

The similar analysis of the variations in thickness on Table IV, shows that the greatest variation is due to differences in thickness of the different plates, but there is also a highly significant variation between different elevations within each plate. This is analogous to the crosswise variation in sheet properties which is usually the major difficulty in papermaking. On the other hand, Table IV also shows that the horizontal and random local variations in thickness are both statistically significant. Since the regression line, equation (2), is not acceptable for predicting thickness, this means that the best estimate of thickness at any one point is the last actual thickness reading at that point.

From the practical point of view the most important

results are the estimates of remaining service life for various portions of the vessel as indicated in Table VI. Here it is indicated that if the present corrosion rate continues the minimum allowable thickness at the present operating pressure will be reached in about 3 years at the knuckle radius of the top head. At the same time it is indicated that other portions of the vessel have a much greater life expectancy. It should be pointed out that in calculating the estimate remaining service life the minimum allowable wall thicknesses as listed in Table VI are calculated by the API-ASME code formulas except that they are based on 100% joint efficiency. It was previously agreed with the insurance company that the reduced joint efficiency (see Table I) would apply only to those points which are within 6 in. of a longitudinal seam. It is felt that if any critical areas occur within such locations, repairs could be made within these restricted areas by the stainless steel weld overlay method. Therefore, it seemed appropriate to base the analysis for the whole vessel on 100% joint efficiency. Another exception made in the code formulas is that the central part of the top head, inside the knuckle section, was considered as part of a hemispherical head. This procedure, for which the insurance company has given tentative approval, is not recommended for routine inspection analysis, but it does indicate more realistically the ultimate service life of this portion of the vessel.

While it is not possible to discuss here all the details and implications of the results shown in Table VI, it is worth mentioning that the observed variation in corrosion rates among different plates within the same corrosion zone does not appear to be statistically significant. Furthermore, Table II shows that all the shell plates have the same analysis, and they were in fact from the same heat. Therefore additional data from other vessels will be required before it will be possible to establish exact correlations between steel analysis and corrosion behavior in digesters.

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The Colorimetric Determination of Pentosans in Highly Refined Pulp

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The quantity of pentosans present in highly refined pulp can be determined with good precision using the colorimetric procedure involving aniline and acetic acid. Reliable results are possible because the methylfurfural and hydroxymethylfurfural formed during the distillation do not interfere. The color development step is empirical but gives good results when several variables are properly controlled. Pulps containing approximately 0.1, 0.2, 0.4, 0.6, 0.8, and 1.0% pentosans were consistently differentiated using this colorimetric method. The procedure is fairly insensitive to most of the variables—such as strength of hydrochloric acid used, the rate of distillation, the size of the pulp sample used, etc. None of the statements in the literature with regard to the percentage of the furfural collected in the first 360 ml. of distillate apply to highly refined pulp. The same is probably true for all statements on the percentage of the total pentosans which can be converted to furfural. Although proposed for highly refined pulps the method also appears to be as satisfactory as the volumetric or the gravimetric procedure for high-level pentosan pulps.

The pentosan content of pulps is estimated by hydrolyzing a sample of the material with strong boiling acid. The pentosans are converted to pentoses which in turn decompose to furfural (*5*). The furfural is distilled over and determined by one of several suitable methods (*2, 9, 12*). A very excellent description of the various methods used for the estimation of pentosans is given by Dorée (*3*). The historical development of the gravimetric procedure is carefully outlined, and a thorough discussion of the distillation variables is given (*7, 8*). An evaluation of the relatively recent work on a volumetric (*6, 8*) and a gravimetric barbituric acid (*10, 13*) procedure is included. Recently a colorimetric procedure using orcinol has been proposed (*12*).

The color produced by aniline and acetic acid has been used as a qualitative test for furfural and is frequently applied to furfural distillates in order to establish whether furfural is still being produced. Youngburg and Pucher (*14*) and later Stillings and Browning (*11*) described an empirical quantitative method for furfural using aniline and acetic acid. The effect of the variables influencing the color development was further studied by Duncan (*4*), and Adams and Castagne (*1*). The latter authors applied the method to the study of some distillation variables and to the analysis of various materials containing relatively large percentages of pentosans.

The volumetric (T 223) and the gravimetric procedures are not well suited to the analysis of material containing less than 1 to 2% pentosans. Both procedures involve the use of a correction factor and the uncertainty in the results is often equal to or greater than the pentosan value.

The aniline acetic acid method is extremely sensitive to furfural and relatively large amounts of methylfurfural and hydroxyfurfural do not interfere. The application of this colorimetric method to the determination of furfural in highly refined pulps was therefore undertaken.

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THE VARIABLES OF THE ANILINE ACETATE COLOR DEVELOPMENT

Youngburg and Pucher (*14*), Stillings and Browning (*11*), and Duncan (*4*) and Adams and Castagne (*1*) have all made a study of the variables which influence the aniline acetate color development. The method is highly empirical and the color which forms is not perfectly stable. The rate of color development and the stability of color are influenced by a number of factors. The following factors are of significance in the color development using aniline acetate: (1) the quantity of aniline used, (2) the quantity of acetic acid used, (3) the quantity of sodium chloride or other stabilizing salts present, (4) the length of the color development period, (5) the temperature of the solution during color development, and (6) whether the flasks are kept in the light or in darkness during color development.

The investigations cited above have fairly definitely established the importance of each of these variables. The recommended method of Adams and Castagne was taken as a basic procedure, but the method was modified slightly at certain points.

The method was made less sensitive by changing the quantities of aniline and acetic acid used. The Adams and Castagne procedure calls for 10 ml. of aniline and 50 ml. of acetic acid. The procedure finally adapted for this study calls for 3 ml. of aniline and 20 ml. of acetic acid. This gives a less sensitive method so that a greater range of pentosan distillates can be handled. The less sensitive procedure appears to be just as reproducible and covers a range two and a half times as large.

The stabilizing salts used in the procedure below are those recommended by Adams and Castagne; 2 grams of sodium chloride, 0.25 gram of oxalic acid, and 0.5 gram of disodium phosphate per 100 ml.

The intensity of the color increases during the first 30 min., remains essentially constant for the next 50 min., and then begins to decrease slowly. The time selected for the procedure given below was 50 to 60 min.

Adams and Castagne held the temperature during the color development at exactly 20°C. The effect of varying the temperature during color development was tested at two levels of furfural. The results appear in Fig. 1.

There is a slight temperature effect in the furfural color development and any laboratory having a range

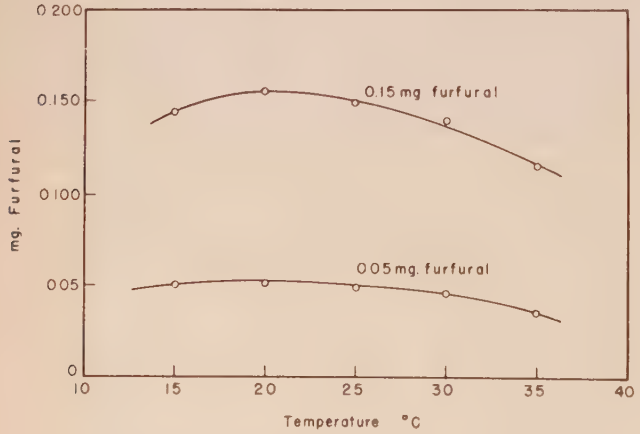

Fig. 1. The effect of temperature on color development

Furfural values were calculated from a standard curve prepared at 25°C.

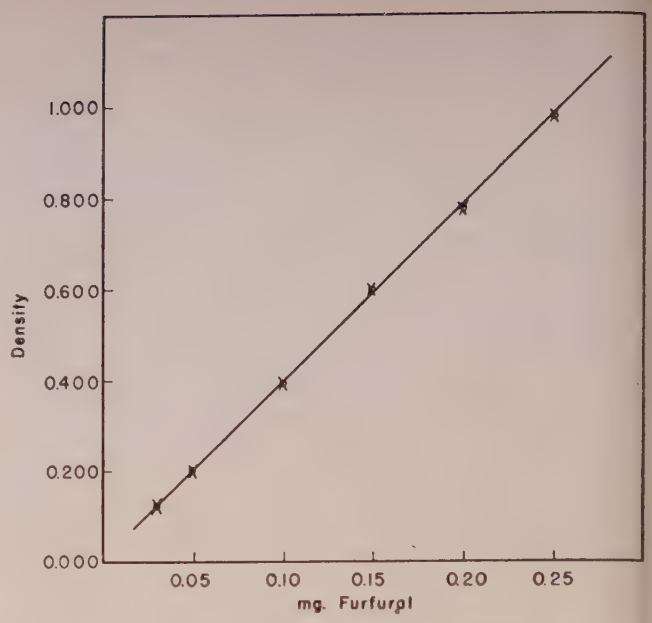

Fig. 2. Effect of light on color development

×—Normal laboratory illumination. ○—Absence of light.

of more than 5° in room temperature should provide some means of approximate temperature control. The experiments of this study were done without temperature control in a room maintained at 25 ± 2°C.

The protection of the flasks from light during color development is less important in the less sensitive procedure used in this study than in the procedure of Adam and Castagne. The difference between the values obtained in complete darkness and those under normal laboratory illumination (east exposure with glass bricks and electric lights on at all times) was negligible. This is shown in Fig. 2. The color was therefore developed for all standard curves and experiments without protection from light.

All of the details of the color development are given in the procedure below.

VARIABLES OF THE DISTILLATION STEPS

After the exact conditions for the colorimetric determination of furfural were fixed the method was used to study the importance of some of the variables of the distillation operation during which the pentosans of the pulp are converted to furfural. For the most part the pulps studied contained 1% or less pentosans. The specific colorimetric method makes possible a detailed study of the rate at which furfural itself is formed since the quantity of methylfurfural and hydroxymethylfurfural formed does not influence the results.

In the most recommended procedures the conditions of the distillation are fixed very rigidly. The following variables are stated or implied to be of importance: (1) the rate of distillation, (2) the total volume of distillate collected, (3) the concentration of the acid (usually hydrochloric acid), (4) the method of adding the acid to the distillation flask, (5) the total volume of acid in the distillation flask, (6) the pulp sample size, and (7) the addition of a salt to the hydrochloric acid during the distillation operation.

Strength of Acid, Rate of Distillation, and Sample Size

Using one highly refined pulp material containing 1 to

Table I. Evaluation of Three Distillation Variables—Percentage Pentosans

Slow distillation									
12% HCl					20% HCl				
Sample size					Sample size				
0.5	1	2	5	10	0.5	1	2	5	10
1.47	1.43	1.33	1.28	1.26	1.56	1.45	1.33	1.25	1.25
1.45	1.43	1.34	1.29	1.28	1.53	1.43	1.34	1.24	1.24

Rapid distillation									
12% HCl					20% HCl				
Sample size					Sample size				
0.5	1	2	5	10	0.5	1	2	5	10
1.38	1.29	1.26	1.22	1.22	1.43	1.38	1.34	1.29	1.29
1.36	1.32	1.26	1.20	1.20	1.45	1.38	1.33	1.28	1.28

1.5% pentosan a group of designed experiments were set up to evaluate three distillation variables: (1) the strength of the hydrochloric acid used, (2) the rate of distillation and (3) the pulp sample size. Twelve and twenty per cent hydrochloric acid were used with 0.5, 1, 2, 5, and 10-gram samples of pulp, using both rapid distillation (360 ml. per 45 min.) and slow distillation (360 ml. per 2 hr.). The flasks used in all distillations of this study were standard taper 500-ml. round bottom flasks. A gas flame was used to furnish heat for the distillation. No wire gauze was used but the flame was applied directly to the portion of the bottom of the flask which protruded through a 3-in. hole in a metal shield. The resulting pentosan values are given in Table I. The changes introduced by these distillation variables were relatively small. The extreme values for averages obtained in this group of experiments were 1.54 and 1.21% pentosans. An analysis of variance showed that there was a large strength of acid, rate of distillation, sample-size interaction. The data were therefore separated on the basis of the strength of acid used and analyzed separately. The two-way Tables II and III contain these data in the form of averages.

The data of the two-way tables show that the average level obtained using 12% hydrochloric acid differed only slightly from the average level using 20% acid. Sample size was significant at both 12 and 20% acid and proved to be more important than the other two factors. Speed of distillation was a significant variable of both 12 and 20% acid, but at 20% acid the changes introduced by varying the sample size are not consistent at the two rates of distillation.

Since pentosan results on highly refined pulps will ordinarily be reported to only 2 significant figures it is apparent that if the sample size is fixed there are no changes of any practical importance introduced by fairly wide variations in speed of distillation or strength of acid. However, these latter factors should not be allowed to vary between extreme limits. These conclusions are all based on the results using one highly refined pulp. The importance of speed of distillation was further evaluated for a large number of highly refined pulps in the experiments reported in Table VII.

Changes in the Concentration of Hydrochloric Acid

The concentration of hydrochloric acid used for the distillation of the pulp sample is usually rigidly fixed at 12%. As the distillation proceeds, the acid quickly goes toward a constant boiling mixture 20.3% hydrochloric acid. The speed with which the acid changes in concentration is shown in the curves of Fig. 3. In three of the experiments the hydrochloric acid was added, as in T 223, by a continuous dripping technique at the rate of 30 ml. every 10 min. In the fourth case the hydrochloric acid was added portion-wise, 30 ml. every 10 min. The concentrations of hydrochloric acid used were 12, 16, and constant boiling or 20.3%.

One set of curves shows the course of the temperature changes in the liquid phase of the various experiments. The other set of curves shows the changes which occurred in the temperature of the vapor phase in each flask.

A few determinations were made of the actual strength of the residual acid in the flask at two to three points in each distillation. These acidity values are given in Table IV.

From the curves of Fig. 3 it is clear that the temperature of the hydrochloric acid in contact with the pulp samples is the same during most of the distillation regardless of the strength of the acid being added. The data of Table IV show that the actual concentration of the acid in the residual liquid of each flask was nearly the same (18 to 20%) during most of the distillation and approached the constant boiling composition (20.3%) fairly closely. The temperature of the vapor in the flasks over the pulp-acid slurry is nearly the same in all three flasks. This would suggest that the vapors in the different flasks were of the same composition. This is not the case. The vapor and liquid, especially in distillations 2 and 3, are not actually in equilibrium since a liquid of different composition is slowly being dripped into the boiling liquid which is continuously going toward the constant boiling composition. Furthermore the vapors are probably not homogenous.

The factors which determine the rate of decomposition of pentosans to furfural are undoubtedly the temperature and the acidity of the liquid in contact with the pulp during the distillation period. These are approximately the same regardless of the concentration of hydrochloric acid employed, in the range of 12 to 20%.

The concentration of the hydrochloric acid being added to the distillation flask was included as one

Table II. Two-Way Table; 12% HCl

	Sample size					
	0.5	1.0	2.0	5.0	10.0	Av.
Slow distillation	1.46	1.43	1.34	1.28	1.27	1.36
Rapid distillation	1.37	1.30	1.26	1.21	1.21	1.27
Av.	1.42	1.37	1.30	1.25	1.24	1.32

Table III. Two-Way Table; 20% HCl

	Sample size					
	0.5	1.0	2.0	5.0	10	Av.
Slow distillation	1.54	1.44	1.34	1.24	1.24	1.36
Rapid distillation	1.44	1.38	1.34	1.29	1.29	1.34
Av.	1.49	1.41	1.34	1.26	1.26	1.35

variable in the designed experiments summarized above, and was shown to be of minor significance. The data of Fig. 3 and of Table IV offer a good explanation for the slight significance of this variable. This was brought out by Dorée (*3*) in his review of the pentosan method. Although there is no justification for insisting on the use of 12% acid, there is likewise no advantage in going to higher acidities. The greater the concentration of hydrochloric acid used, the higher the acidity of the distillate. The neutralization of this acid becomes a problem, especially in those cases where a large aliquot portion of the distillate must be taken for the colorimetric analysis. In any new procedures for pentosans in highly refined pulps it would be as satisfactory to select 12% acid with either a portion-wise or a drop-wise technique for adding the acid.

The Volume of Distillate Collected

Most pentosans procedures recommend collecting exactly 360 ml. of distillate. However, the present revision of the TAPPI procedure for pentosans in pulp, T 223, calls for the collection of only 300 ml. It is generally assumed that the first 300 to 360 ml. of distillate contains virtually all the furfural. This quantity of furfural, however, represents only approximately

Table IV. Percentage HCl in Liquid During Distillation

Type of distillation	*% HCl in flask during distillation* — *35 min.*	*39 min.*	*95 min.*	*99 min.*
Portion-wise addition of 12% HCl; 30 ml./10 min.	18.7[a]	19.4[b]	19.1[a]	19.4[b]
Drop-wise addition of 12% HCl; 30 ml./10 min.	18.6	...	19.3	...
Drop-wise addition of 16% HCl; 30 ml./10 min.	19.8	...	19.8	...
Drop-wise addition of 20.3% HCl; 30 ml./10 min.	20.0	...	20.2	...

[a] Five minutes after adding a 30-ml. portion.
[b] After 39 and 99 min., or 9 min. after adding a 30-ml. portion.

80% of the theoretical amount. The remaining 20% of the pentosans is assumed to be dissipated in side reactions or for one reason or another not convertible to furfural. It is further assumed that additional distillation beyond the first 300 to 360 ml. yields only methyl, or hydroxymethylfurfural. Based on the data obtained on eight different pulps, Launer and Wilson (*8*) concluded that "only in the case of two pulps containing relatively large quantities of pentosans, samples G and H, does it appear that small quantities of pentosans 0.2% remained unconverted after 300 ml. of distillate and that even in these examples the fractions in the second distillate are essentially the same as in the corresponding one of the other pulp."

Using five different pulps the amount of furfural produced in each of five to seven successive 360-ml. portions of distillate was determined. Except where

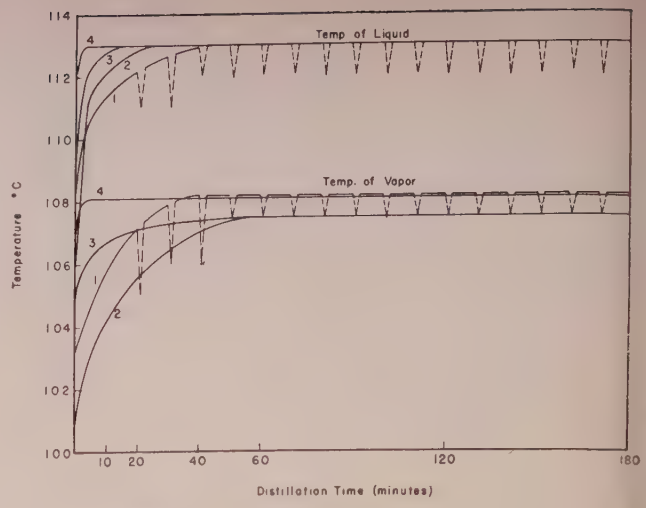

Fig. 3. Temperature change of liquid and vapor during distillation

1. Portion-wise addition of 12% HCl. 2. Drop-wise addition of 12% HCl. 3. Drop-wise addition of 16% HCl. 4. Drop-wise addition of 20.3% HCl.

noted in the table 2-gram samples were distilled with 12% hydrochloric acid using the dripping technique of T 223. The distillations were rapid, 360 ml. every 45 min., except in the one experiment noted. The amount of furfural obtained in each distillate is given in Table V. The table also contains values showing what percentage of the total furfural produced in five to seven portions, was obtained in each portion.

From the data of Table V it is clear that a distillation of 360 ml. does not remove all of the furfural from highly purified pulps. Furthermore, the percentage of furfural coming over in the first 360-ml. portion varies with the level of pentosans in the pulp. For the most highly refined pulps the percentage removed in the first 360 ml. was only 55 to 60% of the total collected in six to seven such portions. In less highly purified pulps the percentage obtained in the first 360 ml. is approximately 85. The percentage in the last portion of distillate is usually as low as one to three.

The relation of these recovery percentages to the percentage yield values of the literature (*8*, *3*, *6*) is not clear. The yield of furfural from xylose is reported to be 88%, from arabinose 74%, and an average yield for the pentosans of pulp is frequently taken as 80%. Previously it was generally assumed that all the furfural that can be recovered distills over in the first 360 ml. and the rest of the furfural is in some way permanently lost. Furthermore it is assumed that the furfural removed in the first 360 ml. represents 74, 80, or 88% of the theoretical amount depending upon the type of pentosans being treated. This picture is not entirely true for

Table V. Furfural in Successive Portions of Distillate

Expt. No. and pulp	*Furfural in each fraction of distillate* — *1st 360 ml.* Mg.	%	*2nd 360 ml.* Mg.	%	*3rd 360 ml.* Mg.	%	*4th 360 ml.* Mg.	%	*5th 360 ml.* Mg.	%	*6th 360 ml.* Mg.	%	*7th 360 ml.* Mg.	%
1-A	84.9	84.1	11.45	11.3	2.3	2.3	1.1	1.1	0.6	0.6	0.4	0.4	0.3	0.3
2-B	52.6	79.2	9.1	13.7	2.1	3.2	1.2	1.8	0.7	1.0	0.4	0.6	0.3	0.5
3-D	13.6	76.1	1.6	9.0	1.0	5.5	0.7	4.0	0.6	3.1	0.4	2.3	...	...
4-D[a]	16.7	82.9	1.8	9.0	1.1	5.4	0.3	1.6	0.2	1.1	...	...	...	...
5-D[b]	13.4	83.1	1.4	8.5	0.5	3.3	0.4	2.3	0.3	1.7	0.2	1.2	...	...
6-F	5.2	58.8	1.4	16.3	0.7	8.4	0.5	5.3	0.4	4.2	0.3	3.6	0.3	3.3
7-G	3.6	59.4	0.8	13.4	0.5	7.8	0.4	5.9	0.31	6.1	0.26	4.3	0.25	4.1

[a] The first 360 ml. were removed in 2 hr., subsequent 360-ml. portions in 45 min. each.
[b] Twenty per cent HCl used rather than 12% as in all other experiments of this table.

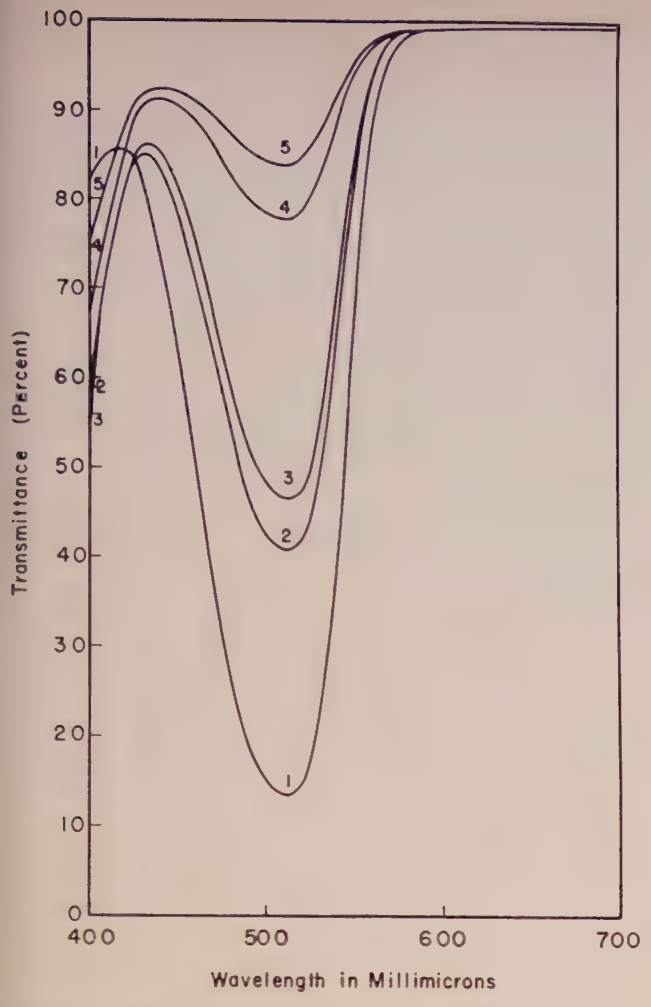

Fig. 4. Spectral transmittance; five successive portions of distillate; aniline color

1. 6 ml. of 1st 360-ml. portion. 2. 25 ml. of 2nd 360-ml. portion. 3. 40 ml. of 3rd 360-ml. portion. 4. 40 ml. of 4th 360-ml. portion. 5. 40 ml. of 5th 360-ml. portion.

highly refined pulps since significant amounts of furfural can be collected after the first 360 ml. have been removed. Another possibility would be that all the pentosans of refined pulps can be converted to furfural provided a sufficiently long distillation period is chosen. The true picture might be something in between these views. That is, the first 360 ml. contain only 60 to 85% of the furfural which can be obtained by longer distillation, but still the maximum which can be removed probably does not represent a 100% yield, based on the total pentosans present.

Nonpentosan constituents of pulps might yield small amounts of furfural. Uronic acids (*3, 9*) yield furfural and carbon dioxide under the conditions of the distillation. Oxycellulose and even pure glucose (*9*) might produce traces of furfural. It is possible that this could account for some of the furfural obtained in the second to seventh 360-ml. portion of distillate.

Until more work is done it would be a mistake to approve any of the previously recommended factors in calculating the pentosan value for highly refined pulps. One possible tentative procedure would be to distill over a total of 1500 to 2000 ml. of liquid and determine the total furfural produced. Then without making any assumptions about the percentage conversion divide this value by 0.727, which is the theoretical conversion factor for pentosans to furfural. In view of the data presented below no correction factor need be applied to compensate for the presence of methylfurfural or hydroxymethylfurfural since they do not interfere with the colorimetric procedure. Another possibility would be to collect 360 ml. of distillate and then make the arbitrary assumption that 80% of the total furfural has been collected.

Interference from Hydroxymethylfurfural

The values reported in Table V assume that there is no interference from methylfurfural or hydroxymethylfurfural in any of the distillates. Browning states that "any error introduced by methyl or hydroxymethylfurfural is less than 1% if the concentration is less than that of furfural" (*11*). The apparent furfural level in the fifth 360-ml. portion of distillate is approximately 0.3 mg. The concentration of methylfurfural and/or hydroxymethylfurfural is unknown and could possibly greatly exceed the level of furfural present. A spectrophotometric curve of the aniline color produced from each of five 360-ml. portions of distillate was therefore made and these are represented graphically in Fig. 4.

From the shape of these curves it is evident that, although there is methylfurfural and/or hydroxymethylfurfural in each fraction of the distillate, furfural is unmistakably present in all portions of the distillate.

Using the absorption at 400 mmu as a measure of the quantity of the methyl or hydroxymethylfurfural present the approximate levels of these impurities in each 360-ml. portion of distillate were calculated. These figures are given in Table VI. This is admittedly an inefficient wavelength at which to measure the absorption due to these impurities, but it does establish

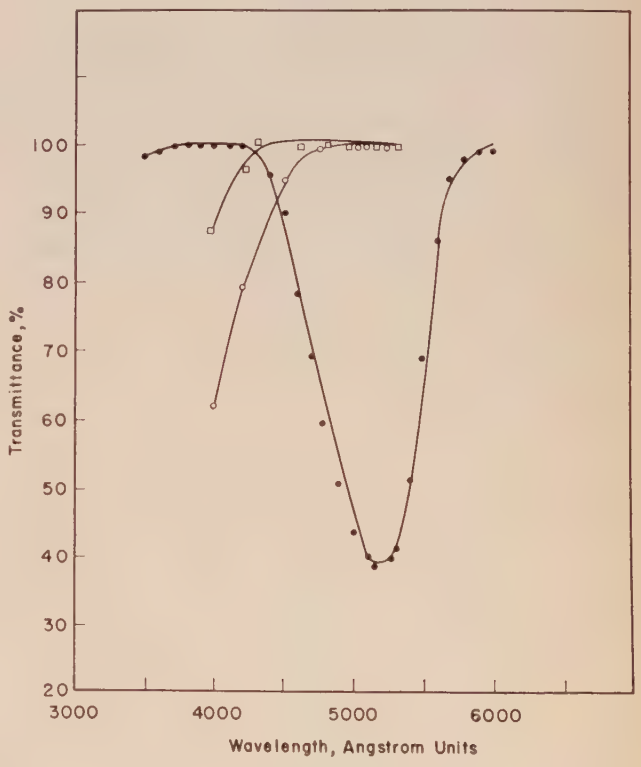

Fig. 5. Spectral transmittance of furfural; methyl furfural; and hydroxymethylfurfural; aniline color. Adams and Castagne (1)

● Furfural. ○ Hydroxymethylfurfural. □ Methylfurfural.

Table VI. Rate of Formation of Methyl and Hydroxymethylfurfural During Distillation

Portion of distillate	*Approximate hydroxymethylfurfural, mg.*	*Furfural, mg.*
1st 360 ml.	1.1	16.7
2nd 360 ml.	0.8	1.8
3rd 360 ml.	0.6	1.1
4th 360 ml.	0.4	0.3
5th 360 ml.	0.3	0.2

the approximate maximum value for their concentration. In almost every case the amount of these impurities is much lower than that of furfural. Therefore, no interference from them was encountered in the aniline acetate procedure and the furfural values are valid. Based on the spectrophotometric curves given by Adams and Castagne, Fig. 5,* it is evident that very much larger quantities of methylfurfural and/or hydroxymethylfurfural could be present without resulting in any interference at 515 mmu.

APPLICATION OF METHOD TO REFINED PULPS

The colorimetric method was used to determine the pentosan content of several highly refined wood pulps and cotton linters, most of which contained less than 1.5% pentosan. Two pulps having a higher pentosan content were also included. The identification and description of the pulps is as follows:

Pulp A was a bleached kraft northeastern spruce.
Pulp B was a semibleached alkali refined kraft northeastern spruce.
Pulp C was a bleached Northeastern hardwood pulp, viscose grade.
Pulp D was an acetylation grade, hot alkali treated sulphite, Western hemlock.
Pulp E is unbleached cotton linters.
Pulp F is cold strong alkali extracted sulphite pulp—Sweden.
Pulp G is a bleached cotton linters acetylation grade.
Pulp E is cold strong alkali extracted acetylation grade cotton linters.

The distillation was limited to 360 ml. in every case, but the rate of distillation was varied. Some were carried out at the rate of 360 ml. in 2 hr. and others 360 ml. in 45 min. A 2-gram sample was used in all experiments. The data of the third column represent a slow distillation starting out with 420 ml. of acid in the flask. The distillation was continued without the addition of more acid until 350 to 360 ml. was collected. All distillations were made in duplicate. The furfural in the distillates was determined using the aniline acetic acid method. All the pulps were also analyzed using the volumetric method T 223 and a gravimetric (phloroglucinol) procedure. The results are given in Table VII.

The pentosan content was calculated from the weight of furfural using the same factor for all three methods. The percentage pentosans converted to furfural was arbitrarily taken as 80.

Volumetric:

$$\text{ml. of 1 } N \text{ } Na_2S_2O_3 \times 0.048 \times \frac{100}{0.727 \times 0.80 \times S} = \% \text{ pentosans}$$

Gravimetric:

$$(W + 0.0052) \times F \times \frac{100}{0.727 \times 0.80 \times S} = \% \text{ pentosans}$$

Colorimetric:

$$\text{wt. furfural} \times \frac{100}{0.727 \times 0.80 \times S} = \% \text{ pentosans}$$

S = weight of pulp sample
F = proper gravimetric factor phloroglucide to furfural
0.048 = grams furfural equivalent to 1 ml. 1 N $Na_2S_2O_3$
0.727 = conversion factor; pentosan to furfural

The pentosan values obtained by the colorimetric method are lower in every case than those by the volumetric or gravimetric procedure. The results do not differ by a constant factor. The percentage difference is greatest for the highly refined pulps where the ratio of furfural to methyl or hydroxymethylfurfural in the distillate is lowest.

The precision of the colorimetric method is very satisfactory. Highly purified pulps having very low levels of pentosan can be consistently distinguished and arranged in order of increasing pentosan content.

Table VII also contains data showing the effect of rate of distillation using eight different pulps. As shown in a preceding paragraph the rapid distillation consistently produced lower pentosan values. The distillation rate need not be controlled rigidly but should not vary as widely as in these experiments.

A set of very satisfactory pentosan values was obtained for these eight pulps by starting the distillation with 420 ml. of 12% hydrochloric acid plus the pulp sample and continuing the distillation without the ad-

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Table VII. Pentosan Values of Several Refined Pulps

Pulp sample	*Colorimetric* 360 ml./2 hr., continuous addition HCl	*Colorimetric* 360 ml./2 hr., no HCl added	*Colorimetric* 360 ml./45 min., continuous addition HCl	*Volumetric* T 223 300 ml./2 hr.	*Gravimetric* 360 ml./2 hr.
A	9.37	9.30	8.91	10.31	10.45
	9.30	9.30	8.98	10.41	10.60
B	5.34	5.27	4.95	6.06	6.29
	5.34	5.27	4.88	6.16	6.24
C	2.86	2.86	2.61	3.64	3.74
	2.86	2.86	2.66	3.70	3.48
D	1.25	1.21	1.12	2.07	1.75
	1.26	1.22	1.12	1.98	1.70
E	1.13	1.25	1.02	1.70	1.40
	1.17	1.16	0.99	1.65	1.40
F	0.64	0.58	0.51	1.28	1.18
	0.66	0.57	0.52	1.12	1.24
G	0.35	0.32	0.28	0.80	0.58
	0.35	0.33	0.28	0.76	0.62
H	0.14	0.13	0.13	0.90	0.54
	0.14	0.14	0.13	0.98	0.60

dition of more acid until 350 to 360 ml. of distillate were collected.

PROCEDURE

The following procedure is tentatively proposed for highly refined pulps. The variables have all been fixed from the information obtained in this study. Some factors are of minor importance and others are more significant. Some statements on the importance of the variables are included in the write-up. This will guard against rigidly fixing an unimportant variable at a value which might later prove to be inconvenient.

Introduce 1 gram of pulp into a distilling flask fitted with a dropping funnel (sample size fairly significant). Add 150 ml. of 12% hydrochloric acid to the flask and add acid of the same strength to the dropping funnel (strength of acid used in the range 12 to 20% of minor significance). Distill at the rate of approximately 60 ml. every 10 min. (rate fairly significant) and collect 360 ml. of distillate (the volume of distillate is very important, and is arbitrarily fixed at 360 ml. until more work is done on problem). Dilute the distillate to 500 ml. in a volumetric flask, stir thoroughly.

Using a pipet transfer a 1 to 25 ml. aliquot portion (depending on the pentosan content of the pulp and the cell length of colorimeter) to a 100-ml. volumetric flask. Add 1 drop of phenolphthalein indicator, bring the volume to approximately 30 ml. with water, and neutralize with 6 *N* sodium hydroxide. Add 10 ml. of 20% sodium chloride, 5 ml. of 5% oxalic acid, and 5 ml. of 10% disodium phosphate. Mix the solution thoroughly after each addition. Add 20 ml. of glacial acetic acid ACS. Mix and add 3 ml. of freshly distilled aniline. Dilute to the mark and blend thoroughly. Allow the flasks to stand at room temperature for 55 to 60 min. If the temperature of the laboratory is not controlled within a 4 to 5°C. range use some means of approximate temperature control. If extreme variations in the illumination of the laboratory are frequently encountered develop the color in the absence of light.

After the standing period, read the optical density on a colorimeter with a 515 mmu filter.

Prepare a standard curve using known amounts of redistilled furfural. If cells of different lengths are available more than one standard curve should be prepared. This will increase the range of samples which can be measured without repeating the color development step.

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Studies in Continuous Alkaline Pulping

II. High Yield Cellulose Pulp from Scrub Oaks

W. J. NOLAN and W. F. BROWN

Further data are presented on continuous pulping involving presteaming and shredding of chips followed by pulping at substantially constant liquor concentration and steam pressure. The wood pulped was scrub turkey oak, processed for the purpose of obtaining high alpha-cellulose, low pentosan pulps with no attempt to produce high physical strength. Liquor concentrations from 20 to 80 grams per liter, steam pressures from 115 to 170 p.s.i.g. and cooking times from 10 to 40 min. were studied. A definite relationship between concentration of cooking liquor and per cent pentosan in pulp has been found. Concentrations of 80 grams Na_2O per liter will produce pulps containing 2 to 3% pentosan while concentrations of 20 grams per liter will produce pulps of 9 to 11% pentosan. Steam pressure and cooking time have only a slight effect on pentosan content. Presteaming for 10 min. at 170 p.s.i.g. steam pressure in dilute sodium carbonate solution removes about 25% of the pentosan in the original chips. The ratio of pentosan removed to total wood removed by alkaline presteaming appears to be about the same as in acid steaming at 4.5 pH. Yield, lignin, pentosan, and alpha cellulose percentages in pulp may be closely controlled by specification of the pulping variables. A pulp of 45.3% yield, 2.6% lignin, 92.2% alpha, and 8.8% pentosan can be produced in 15 min. at 148 p.s.i.g. and 35 grams per liter. Another pulp of 40.0% yield, 1.5% lignin, 96.6% alpha, and 2.2% pentosan can be produced in 10 min. at 148 p.s.i.g. and 80 grams per liter. The alpha-cellulose content may be controlled closely at any value between 85 and 95%, depending on conditions used. It is estimated that 90% of the alpha-cellulose in the original wood may be recovered in the pulp. No data on viscosity D.P. numbers or alkali purification are included in this study. Pulps sent to a rayon manufacturer were reported to have a D.P. number of 1250 after bleaching, which reduced to 990 after further purification with hot alkali. The data indicate that turkey oak can be readily converted into high grade dissolving pulp by continuous cooking.

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The results of work previously reported (*1*)

showed that pulps of very high alpha-cellulose and low pentosan content could be produced in very short cooking times by continuous pulping methods. These pulp characteristics indicated a very promising application in the field of dissolving pulps. Such an application seemed particularly worth investigation in the hardwood field where the typical short fiber lengths are unsuited for high strength paper pulps but present no problem in the dissolving field.

Heretofore low pentosan content in pulp has been produced by acid hydrolysis prior to alkaline pulping. The prospects of producing such pulps in a completely alkaline continuous process of short duration without having to resort to acid hydrolysis offer several attractions. Considerably higher yields of pulp should result since the dissolution of cellulose which accompanies the removal of pentosan by acid hydrolysis is eliminated. None of the reduction in cellulose chain length inherent in the acid hydrolysis step occurs and a pulp of more uniform D.P. number results. The uniform mixing of chips and liquor which is characteristic of continuous pulping further promotes the production of a pulp of uniform D.P.

The work mentioned in the first paragraph indicated that highly sustained concentrations of alkali might produce pulps of very low pentosan content. For example, pulps produced at 35 grams per liter contained 9.0% pentosan while those cooked with 50 grams per liter resulted in a pentosan content of 5.4%. This is in contrast to hardwood pulps produced by conventional batch methods containing 18 to 20% pentosan. It should be possible to use even higher concentrations of alkali for further reduction of pentosan content. The decrease in yield which normally accompanies stronger cooking liquors can be prevented by either decreased cooking time or decreased temperature.

References in the literature to investigations into the production of high purity pulps by alkaline pulping alone are very meager. Nearly all such studies involve the use of acid hydrolysis prior to alkaline pulping. Great masses of data are reported in which pentosan content of pulps produced at various initial concentrations are given. Almost invariably, however, liquor-wood ratios were held at the conventional low values resulting in low alkali concentration very early in the cook. These low liquor ratios masked any possible relationship between pentosan content of pulps and alkali concentration in the liquor.

The data of Schwartz and Bray (*2*), in which liquor-wood ratios were varied from normal low values up to values of 40 and 60, showed that increased sustained concentration caused decreased pentosan content. Pentosan contents of 6.0 to 6.7% were realized at a liquor-wood ratio of 15 while values of 3.5 to 4.0% were reported at a ratio of 60. In all cases initial liquor concentration was 60 grams per liter. Unfortunately, low pentosan values were accompanied by low yields, in the neighborhood of 37 to 38%.

Meller (*3*) found that pulps of high alpha-cellulose and low pentosan content could be obtained from pine chips by the use of high percentages of alkali at low liquor-wood ratios. Pulping was carried out in both one and two-stage cooking. Pulps containing 91 to 93% alpha-cellulose and 3 to 4% pentosan were produced in two-stage pulping. Sufficient chemical was present so that final concentration of alkali in the cooking liquor amounted to 4.7 to 7.7% alkali. Pulp yields were kept at a surprisingly high level, probably due to the low steam pressures, 46 to 80 p.s.i.g., employed. The work indicated that concentration of alkali, much more than cooking temperature, might control the pentosan content of the resultant pulps.

It was decided, in view of the above considerations, to carry out this investigation in such a manner that any relationship between alkali concentration and pentosan content of pulps could be evaluated. Concentration of alkali should be held substantially constant throughout each cook. Concentrations to be studied were set between the limits of 20 and 80 grams per liter active alkali, expressed as Na_2O. This wide range of concentration should produce great differences in reaction rate and corresponding differences in pulp yield. In order to counteract the effect of these concentration changes with compensating changes in cooking pressure or reaction time, it becomes necessary to evaluate the effect of such changes. It was decided to study each concentration in the range of 115 to 170 p.s.i.g. steam pressures and reaction times between 10 and 30 min.

It had been planned, in laying out the research program, to investigate alkali purification of pulps and effects of the pulping variables on D.P. number, viscosity, and other pulp characteristics of particular interest to the consumers of dissolving pulps. This planning was based on the expectation of a considerable increase in laboratory personnel. Since this increase has not occurred as yet, it was necessary to curtail the last phase of the study. These details will be reported at a later date.

METHOD OF PROCEDURE AND PRESENTATION OF DATA

Scrub oak logs, 3 to 8 in. in diameter (with bark on) were cut in 5-ft. lengths. Their bark was removed by the chain debarker in the laboratory. Peeled logs were converted into commercial size chips by processing them through the chipper of the Hudson Pulp and Paper Co. at Palatka, Fla. Chips were screened on the laboratory screen and the fraction passing through 1-mesh and retained on 2-mesh was air-dried and stored for later use. The chips were processed in both the presteaming and pulping stages in equipment described previously (*4*).

Chips were first softened by steaming and their surface areas expanded by hammer milling. The dry chips were charged in the digesters which had already been brought to the full steaming pressure of 170 p.s.i.g. Eleven liters of preheated sodium carbonate solution (4.8 grams per liter) were added to each 200 grams of dry wood charged. At the end of 10 min of steaming at 170 p.s.i.g. digesters were blown to atmospheric pressure. The solution, just prior to blowing, had a pH value of 8.5.

Several additional steaming tests were carried out to determine the effect of the steaming process on the chips. Dry chips were steamed in the sodium carbonate solution at 170 p.s.i.g. steam pressure for periods of 5, 10, and 15 min. Yield and pentosan content of resultant chips were determined. Another batch of chips, which had been soaked in sodium carbonate

Table I. Yield and Pentosan Content of Steamed Chips

(Chips Steamed in Dilute Na_2Co_3—4.8g./1)

Pretreatment	Steam pressure, p.s.i.g.	Steam time, (min.)	Yield of orig. chips, %	Pentosan content, %	Pentosan removed, %
None	...	0	100	18.8	0.0
None	170	5	94.0	16.4	18.1
None	170	10	90.7	15.2	26.6
None	170	15	84.7	13.0	41.5
Chips soaked 72 hr.	170	5	92.3	17.5	14.4
in dilute Na_2CO_3 at	170	10	87.2	16.2	25.0
room temp.	170	15	87.2	15.5	28.2

Per cent lignin in original wood = 28.3; % lignin in chips steamed 10 min. = 31.3%.

Per cent lignin removed in 10 min. $= \frac{(28.3 - 90.7 \times 0.313)}{28.3} \times 100 = \frac{(28.3 - 28.4)}{28.3} 100 = 0.$

solution (4.8 grams per liter) for 3 days at room temperature, were steamed in the same manner as the dry chips. Yield and pentosan values were determined on the processed chips.

The softened chips were transported to another section of the campus for mechanical treatment. By the time treatment occurred the chips had cooled to room temperature but had been softened to a tough leathery condition. Mechanical treatment consisted of a single pass through a 5-hp Sturtevant hammer mill with $^1/_2$ in. expanded metal discharge screens. The processed chips retained the same length in the grain direction as the original material but had been split along the grain into cross sections of match stick size or smaller. This material was stored wet (45 to 50% moisture) in airtight cans prior to pulping.

Pulping was carried out at substantially constant concentration and constant steam pressure. In no case did concentration drop by more than 10% of initial concentration. Concentrations studied were 20, 25, 35, 50, and 80 grams per liter active alkali expressed as Na_2O. In all cases 25% sulphidity was used. Steam pressures of 115, 130, 148, and 170 p.s.i.g. were used and cooking times of 10, 15, 20, and sometimes 30 and 40 min. were studied.

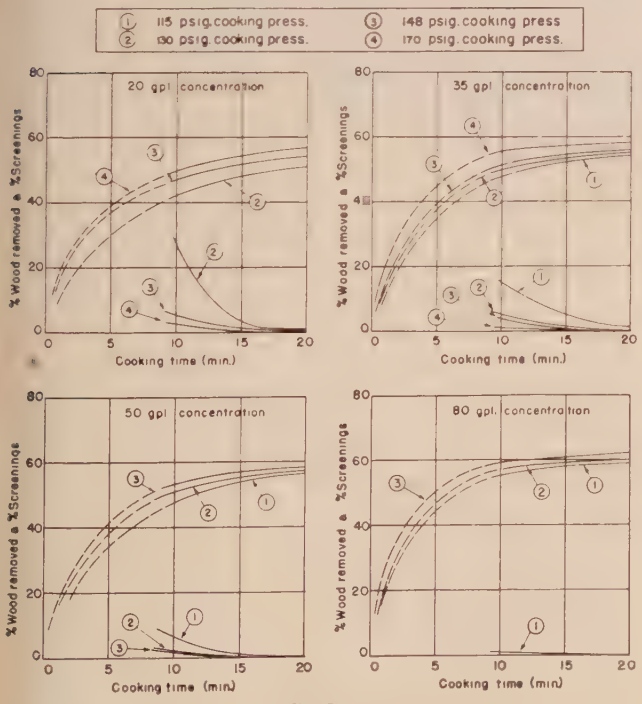
① 115 psig. cooking press.
② 130 psig. cooking press.
③ 148 psig. cooking press
④ 170 psig. cooking press.
20 gpl concentration
35 gpl concentration
50 gpl concentration
80 gpl. concentration
% Wood removed & % Screenings
Cooking time (min.)

Fig. 1. Cooking curves

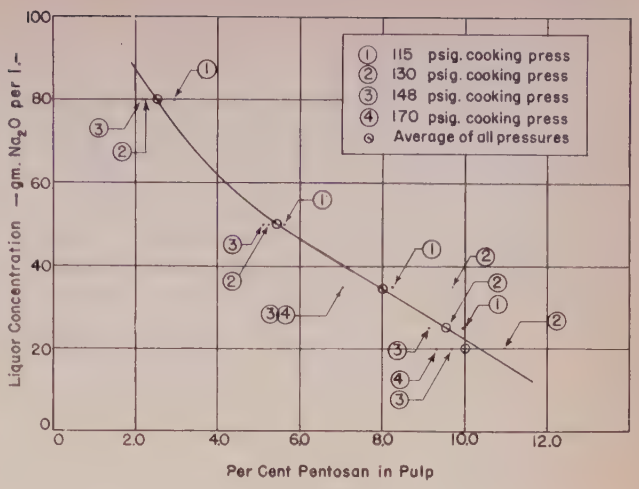
① 115 psig. cooking press
② 130 psig. cooking press
③ 148 psig. cooking press
④ 170 psig. cooking press
⊙ Average of all pressures
Liquor Concentration — gm. Na2O per l.
Per Cent Pentosan in Pulp

Fig. 2. Pentosan removal with liquor concentration

The design of the experimental pulping equipment was such that it was impossible to maintain concentration by the injection of fortified liquor during the cooks. Sufficient liquor had to be supplied at the start of the cook to insure very slight drop in concentration during the cook. Liquor ratios of 40:1 were used, corresponding to 80% chemical at 20 grams per liter concentration or 190% chemical when 50 grams per liter liquor was used. If it had been possible to maintain concentration by injection of high concentration alkali, normal liquor ratios of 3:1 to 5:1 could have been used with injection of only sufficient alkali to replace that consumed in a normal cook.

Pulps were washed thoroughly with hot water and screened through the laboratory flat screen, using a screen plate with 0.008-in. slots. Yield values were determined in the usual manner and samples were air-dried at room temperature and stored for chemical analysis. Selected pulps were analyzed for lignin, alpha-cellulose, and pentosan. Permanganate numbers and single stage bleach requirements were determined for some pulp. All analytical methods were carried out according to TAPPI standards. No physical strength data were determined.

The effect of chip steaming on yield and pentosan content is presented in Table I. Table II contains the yield and analytical data for pulps cooked under the complete range of concentration, steam pressure, and cooking time. In all cases yield and analytical data are averages for at least two cases. A total of 154 cooks were carried out to produce the results listed in Table II.

Yield and screenings data from Table II are presented graphically in Fig. 1. Per cent wood removed and per cent screenings in pulp are plotted against cooking time. Figure 2 shows a definite relationship between pentosan content of pulps and concentration of chemical in the cooking liquor. The usual relationship between per cent lignin removed and per cent wood removed is shown in Fig. 3. In Fig. 4 bleach requirements of pulps are plotted against corresponding permanganate numbers.

DISCUSSION

Effect of Chip Steaming

The data in Table I give an indication of what is

occurring in the preliminary steaming process. Chips were steamed in dilute sodium carbonate solution at 170 p.s.i.g. for periods of 5, 10, and 15 min. The data show that yield of chips decreases as time increases. An investigation of lignin and pentosan content showed that no lignin is removed but pentosan is removed quite rapidly. About half of the total material that is dissolved consists of pentosan. At the end of 15 min. steaming time, 41.5% of the total pentosan is dissolved.

It was thought that this rapid removal of pentosan might be due to acid hydrolysis. Organic acids might be generated in the center of the chips before the dilute carbonate has had a chance to counteract them. Another batch of chips was soaked for 72 hr. in the same dilute sodium carbonate prior to steaming to check this theory. The chips were drained and then steamed in exactly the same manner as the original dry chips. It was found that less pentosan was removed when carbonate was present at the center of the chip, particularly in the 15-min. steaming, indicating that some pentosan is being removed by acid hydrolysis. However, acid hydrolysis cannot account for the removal of most of the pentosan which is dissolved by very dilute alkali (pH 8.0 to 8.5) at 170 p.s.i.g. steam pressure. The actual temperature during the steaming process varied from 350°F. at the start to about 368° at the end of 15 min.

Yields of steamed chips appear to be lower when the chips are soaked in carbonate before steaming. The yield figure for the 15-min. steaming of presoaked chips is probably somewhat in error as the trend indicates it should be lower than the 87.2% recorded. This decrease in yield may be accounted for by the material which is removed in the extended soaking period. About 2.5% of the wood is dissolved by soaking.

A batch of dry chips was steamed 10 min. at 170 p.s.i.g. using water as the steaming medium instead

Table II. Cooking Data and Chemical Analysis of Pulps[a]

Liquor concentration, g./l.	*Cooking pressure, p.s.i.g.*	*Cooking time, min.*	*Yield, % of steamed wood*		*Screenings, % of pulp*	*Analysis of pulp*				
			Total	*Screen*		*Lignin, %*	*Alpha-cellulose, %*	*Pentosan, %*	*Bleach factor*	*Permanganate no.*
20	130	10	57.2	40.8	28.5	15.1	..	11.0	..	..
20	130	15	51.2	49.6	3.3	10.9	..	11.1	..	..
20	130	19	49.2	48.6	1.2	8.3	..	10.8	..	..
20	148	10	50.6	47.9	5.4	6.6	..	10.0	..	..
20	148	15	47.4	46.6	1.4	3.3	..	9.5	..	..
20	148	20	46.1	45.8	0.6	3.0	..	9.7	..	..
20	148	30	44.8	44.7	0.2	2.3	..	9.4	..	..
20	170	10	50.2	49.0	2.4	7.2	..	9.6	..	..
20	170	15	46.0	45.7	0.6	4.5	..	9.0	..	..
20	170	20	43.0	42.9	0.2	4.0	..	9.2	..	..
25	115	10	54.4	44.7	18.2	..	..	..	..	32.4
25	115	15	50.6	49.0	2.3	8.6	86.7	10.0	..	21.8
25	115	20	47.3	46.8	0.9	6.4	89.0	9.9	8.9	18.5
25	115	30	45.6	45.5	0.3	..	..	..	..	..
25	130	10	50.6	48.0	5.0	..	..	..	..	..
25	130	15	47.8	47.1	1.3	5.0	88.8	9.6	11.8	17.6
25	130	20	46.3	46.1	0.4	4.1	..	9.6	11.5	14.6
25	130	30	44.5	44.4	0.1	..	..	..	..	..
25	148	10	48.4	46.8	3.2	6.9	87.5	9.4	13.2	22.1
25	148	15	45.2	44.8	0.8	4.3	87.4	8.9	8.3	13.8
25	148	20	43.6	43.6	0.1	..	..	..	..	..
25	148	30	43.2	43.2	0.1	..	..	..	..	..
35	115	10	51.6	43.9	15.0	8.6	..	8.4	..	..
35	115	15	49.3	47.0	4.6	..	..	..	10.6	..
35	115	20	45.6	45.4	0.4	3.3	..	8.0	..	..
35	130	10	49.6	47.2	4.8	7.2	..	8.8	..	..
35	130	15	46.6	45.9	1.5	6.2	87.9	10.6	..	..
35	148	10	48.2	46.6	3.2	4.2	..	7.7	..	..
35	148	15	45.3	45.0	0.6	2.6	92.2	8.8	4.7	9.7
35	148	20	43.4	43.3	0.0	1.7	92.8	6.8	4.3	8 4
35	148	30	40.7	40.7	0.0	1.3	94.4	5.8	2.5	7.3
35	148	40	39.8	39.8	0.1	1.2	93.1	5.8	2.4	7.6
35	170	10	44.0	43.7	0.8	1.9	..	6.5	..	..
35	170	15	42.3	42.1	0.6	1.9	92.5	7.4	4.5	..
50	115	10	51.4	48.1	6.5	..	..	..	..	..
50	115	15	45.2	44.9	0.8	4.0	90.8	5.7	5.7	16.0
50	115	20	43.3	43.2	0.2	2.9	94.7	5.5	..	11.3
50	130	10	47.5	46.9	1.3	5.7	..	6.5	..	..
50	130	15	44.0	43.8	0.4	8.4	91.7	4.0	6.2	13.2
50	148	10	45.8	44.8	2.2	4.0	..	5.8	..	..
50	148	15	42.2	42.0	0.4	2.2	93.8	5.3	5.3	..
50	148	20	41.2	41.1	0.2	1.4	..	4.5	..	..
80	115	10	43.8	43.4	0.8	7.5	93.0	3.0	8.0	16.1
80	115	15	41.6	41.3	0.8	3.5	93.4	2.9	..	..
80	115	20	40.5	40.5	0.0	..	..	..	..	..
80	115	30	39.9	39.9	0.0	..	..	..	..	..
80	130	10	42.2	42.0	0.6	2.6	88.3	2.2	..	..
80	130	15	40.4	40.4	0.1	2.5	94.0	2.2	..	..
80	130	20	39.5	39.5	0.0	..	..	..	..	..
80	130	30	37.3	37.3	0.0	..	..	..	..	..
80	148	10	40.0	39.9	0.14	1.5	96.6	2.2	..	..
80	148	15	38.8	38.8	0.0	1.9	94.4	2.1	..	..
80	148	20	37.0	37.0	0.0	..	..	..	..	..
80	148	30	34.4	34.4	0.0	..	..	..	..	..

[a] A liquor-wood ratio of 40:1 was used in all cooks.

of alkali. Final pH was 4.0. Yield was quite low, 82.8% of the original wood, and 65.3% of the pentosan was removed. Pentosan removed constituted 52.2% of total material dissolved. In the case of the 10-min. steaming in alkaline solution the pentosan removed amounts to 53.7% of the total material dissolved. These figures indicate that removal of pentosan by acid hydrolysis offers no relative advantage over alkaline removal. In fact, the acid hydrolysis quite possibly degrades some of the cellulose in the chip without actually dissolving it. This last statement is a conjecture as no data have been compiled to substantiate it.

Effect of the Cooking Variables on Rate of Pulping

The pulping data for all conditions studied are presented in Table II. The yield and screenings data from this table are shown graphically in Fig. 1. The cooking curves in the range of 0 to 10 min. reaction time are shown by dotted lines as the exact shape of the curves in this range is not known. It is unfortunate that no cooking data were taken at cooking times of 5 min. At the time the data were being accumulated it was felt that screenings data at 5 min. reaction time would be too erratic to warrant determination.

The first point to be noted from these curves is the remarkable speed of reaction, causing very rapid removal of woody material and conversion of the shredded chips to screened pulp. This speed of reaction becomes apparent when it is realized that a conventional cooking curve would appear as a straight line through the axis and cutting the line of 20 min. cooking time at about 37% wood removed.

The shape of the wood removal curves appears quite similar to those for conventional batch pulping. Even though the concentration of chemical is just as high at 20 min. cooking time as it was at the start of the reaction, the curves tend to become asymptotic with the time axis. This characteristic is quite similar to the asymptotic appearance of conventional cooking curves at 3 to 4 hr. time when the concentration has dropped to a very low value.

There is definite indication that a certain amount of woody material is easily removed, accounting for the steep slope of the wood removal curve at the start of the cook, and another fraction which is removed slowly regardless of the final concentration. The amount of this latter fraction seems to depend on the temperature and concentration of pulping. This can be seen in Fig. 1 by the relative positions of the wood removal curves at 35, 50, and 80 grams per liter for the different steam pressures.

Figure 1 brings out the extremely rapid rate of screenings reduction inherent in the continuous pulping process. At a concentration of 20 grams per liter, screenings will not be appreciable unless cooking pressure is 130 p.s.i.g. or less. At 35 grams per liter, cooking pressure must be 115 p.s.i.g. or less before screenings become a problem. Above 35 grams per liter none of the pressures studied will produce uncooked pulp to any appreciable degree. The screenings removal curves shown seem noteworthy when it is considered that per cent screenings from a conventional cook would be too high to even appear on the charts shown.

Pulps of the same yield values can be obtained under a wide variety of conditions. For example, a pulp of about 49.5% yield can be obtained at 35 grams per liter, holding the digester at 115 p.s.i.g. for 15 min. or at 130 p.s.i.g. for 10 min. A pulp of 43.4% yield can be produced at 35 grams per liter, 115 p.s.i.g., and 20 min. A pulp of about 42.2% yield will result from 35 grams per liter, 170 p.s.i.g., and 15 min. or from 80 grams per liter, 130 p.s.i.g., and 10 min. Yield and screenings may be duplicated by various combinations of concentration, temperature, and time but duplications of chemical analysis of pulps cannot be arrived at so readily.

Effect of the Cooking Variables on Chemical Analysis of Pulps

It has been found that there is a definite relationship between concentration of cooking liquor and per cent pentosan in the resultant pulp. This correlation is shown to a remarkable degree in Fig. 2, when one considers the wide range of steam pressures and cooking times involved. The nature of the pentosan determination itself might well account for most of the divergence from the overall averages shown. Pentosan data for a total of 45 different pulps were used in the verification of this relationship.

The concentration of the cooking liquor determines the per cent pentosan in the pulp but says nothing about the percentage of the original pentosan which is removed. This quantity is determined by yield of pulp. It is in this respect that steam pressure and cooking time influence pentosan removal. A pulp of high yield with up to about 30% screenings will have practically the same pentosan percentage in its screened fiber as a pulp of low yield and negligible screenings, as long as the same concentration is used in both cooks.

Figure 3 shows the usual relationship between lignin removal and wood removal. In the case of pulping under constant conditions as shown, a definite value of per cent lignin removal will require a higher value of per cent wood removal than will the same lignin value in pulp produced by conventional pulping processes. The high rate of pentosan removal under constant conditions as compared to slight pentosan removal in batch pulping accounts for this change in per cent wood removed.

There is no direct relationship between lignin content of pulps and liquor concentration. Lignin content is more a function of all the cooking variables which affect yield. For example, it is possible, by adjustment of cooking variables, to produce a pulp low in lignin and yield but high in pentosan, or one high in lignin and yield but low in pentosan. It is not possible under the conditions studied to produce a high yield pulp which is low in lignin.

Pulping under constant conditions seems to produce pulps considerably higher in alpha-cellulose than can be obtained by conventional batch methods. Pulps with yields as high as 50% contained about 87% alpha-cellulose. Calculation of total alpha-cellulose in the wood which is recovered in the pulp cannot be done accurately due to the difficulty of alpha-cellulose determination in wood. An estimate of alpha-cellulose in wood was obtained by evaluating alpha-cellulose in the residue from holocellulose determination of wood.

From this calculation the data show recovery of over 90% of original alpha-cellulose in the final pulp.

This high recovery of alpha-cellulose in continuous pulping is the result of two factors. First, the pulping is so rapid and uniform that cellulose degradation by alkali is kept at a minimum. Second, pentosan removal is accomplished by use of alkali, avoiding acid hydrolysis which promotes cellulose degradation.

Bleach requirements, as specified by TAPPI Standard T 219 m, were determined on representative pulps. The corresponding permanganate numbers were also evaluated. A plot of the bleach factors against permanganate numbers is shown in Fig. 4.

The relationship is shown to be highly qualitative. This laboratory has not yet been able to find any close agreement between bleach factor and permanganate number for either pine or the hardwoods.

One pulp was tested in three and five-stage bleaching, using chlorine, caustic extraction, and hypochlorite. The pulp was cooked 15 min. with liquor of 50 grams per liter and 25% sulphidity at 148 p.s.i.g. This pulp, using 2.7% chlorine in three stages, bleached to a G.E. brightness of 85. In five stages the pulp required 2.5 per cent chlorine to the same brightness.

It had been planned to include in this investigation an extensive study of pulp purification by means of chlorine and hot and cold alkali extraction. Lack of laboratory personnel has required postponement of this phase to a later date. Samples of the pulp used for the multistage bleach tests as just described were sent to some of the consumers of high alpha dissolving pulp. The D.P. of the pulp, after bleaching with sodium chlorite, was found to be 1250. After hot alkali purification and bleaching, the D.P. was 995. Laboratory tests showed the pulp to be satisfactory for the manufacture of acetate rayon.

It is hoped that the laboratory staff can be augmented sufficiently in the near future to include pulp purification and determination of constants necessary to high alpha pulps for dissolving and other grades. This phase of the recovery of cellulose from wood is particularly important in view of the flexibility of continuous pulping. Lignin, pentosan, and alpha-cellulose content of pulps from the digester can be easily controlled. It now becomes necessary to evaluate just how much of the ultimate purification should be carried out in the digester and how much should be left to oxidation and

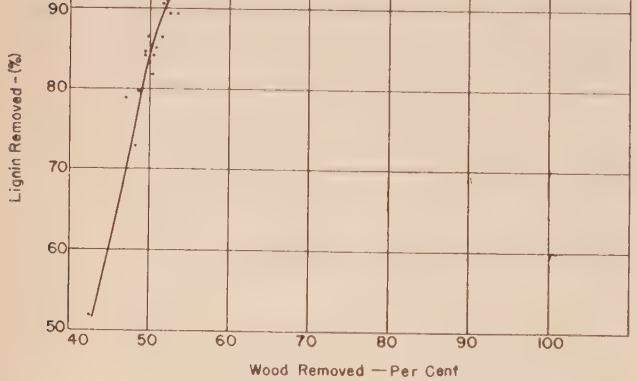

Fig. 3. Original lignin removed vs. per cent wood removed

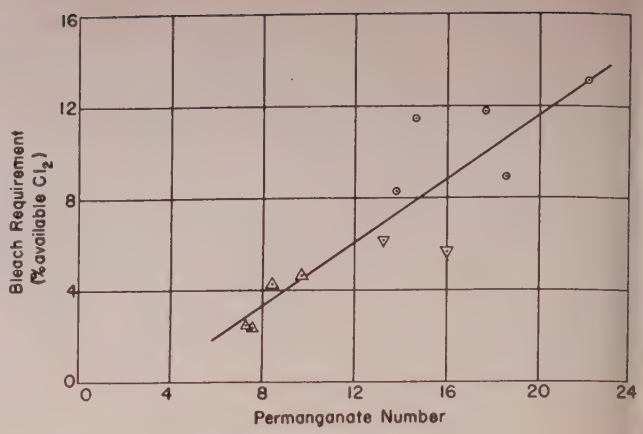

Fig. 4. Bleach factor—permanganate number relationship for pulps

alkali purification. This evaluation will result in the maximum yield of purified cellulose of the proper degree of polymerization, viscosity, filterability, and other characteristics.

CONCLUSIONS

Pulps may be produced from turkey oak with alpha-cellulose content as high as 95% in 15 min. Pentosan content of these pulps can be controlled to values as low as 3% or less. These pulps have a D.P. number of 1250 after bleaching.

The variables of pulping can be so controlled as to produce pulps of the desired yield and analysis under different conditions. As chemical concentration is increased, cooking temperature, and/or time may be reduced to compensate for the accelerating effect of concentration. Increased pulping time permits the use of lower concentration or temperature.

Pulps resulting from continuous pulping of turkey oak are easily bleached, requiring 2.7% chlorine for producing a pulp of 85 G.E. brightness in three stages.

A direct relationship has been found between pentosan content of pulps and concentration of alkali in the cooking liquor. The pentosan content of pulp is only slightly affected by cooking time or temperature at any specific concentration of alkali.

Presteaming of chips in dilute alkali prior to mechanical steaming removes a high proportion of pentosan. Ten minutes' exposure to 170 p.s.i.g. steam results in removal of over 25% of the pentosan in the original wood. About 8 to 9% of the wood is removed in this steaming process.

It is a simple matter to regulate the lignin and pentosan content of pulps over wide ranges by adjustment of the cooking variables. This fact makes it highly desirable to evaluate methods of pulp purification so that a proper balance between pulping and subsequent purification may be set up.

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Chemical Reaction Equilibria in the Combustion of Sodium-Base Pulping Liquors

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This investigation has been devoted to a study of the chemical reaction equilibria in the combustion of sodium-base pulping liquors. Thermodynamic principles were employed to calculate the equilibria in certain basic reactions involving all the compounds which might be expected to exist in a sodium-base system under recovery conditions; from these basic reactions other reactions of interest were derived and their equilibria were computed. As a result of these computations, it has been shown why the usual products are obtained in the kraft recovery process; a new process for the recovery of chemicals in the neutral sulphite or sodium-base acid sulphite process has been suggested; a reason for the high sulphur losses relative to the sodium losses in the kraft process has been demonstrated; and a possible explanation for the explosion difficulties sometimes encountered in the dissolving of a kraft furnace smelt has been suggested.

THREE sodium-base pulping processes are presently in use by the wood pulp industry: the kraft process, the sodium sulphite or neutral sulphite process, and the sodium-base acid sulphite process. Of these, the kraft process is by far the most used by the industry today. Experience has shown that spent kraft liquor can be burned under reducing conditions to yield chemicals (primarily sodium carbonate and sodium sulphide) which can be readily converted into fresh cooking liquor by treatment with lime. This treatment converts the carbonate to sodium hydroxide so that the final liquor contains, for the most part, the desired mixture of sodium hydroxide and sodium sulphide. Experience has also indicated that, under similar combustion conditions, the spent liquor from either the sodium-base acid sulphite process or the neutral sulphite process also yields sodium carbonate and sodium sulphide. However, whereas the formation of sodium sulphide is quite desirable in the kraft process, the formation of this compound is troublesome in the other two processes. In the case of the neutral sulphite process, the objective of recovery is the regeneration of sodium sulphite from the furnace products, whereas the objective of recovery in the sodium-base acid sulphite process is the regeneration of sodium bisulphite. Either of these compounds can be obtained from a solution of sodium carbonate by treating the solution with sulphur dioxide. However, if the solution contains sodium sulphide as well as sodium carbonate, treatment with sulphur dioxide tends to convert the sodium sulphide into sodium thiosulphate which is generally conceded to be an undesirable component of a neutral or acid sulphite liquor.

Thus, although the recovery of spent kraft liquor is in widespread practice today, the recovery of a sodium-base acid sulphite or neutral sulphite spent liquor still presents a problem. In the belief that a basic study of the chemical reaction equilibria in the combustion of sodium base liquors would be of assistance in understanding the problems of recovery, such an investigation was undertaken.

Chemical reaction equilibria can be readily calculated by the application of thermodynamic principles if the necessary thermal data for the compounds involved are known; some of the more important concepts will be discussed below in order to make clear the principles employed in this problem.

Consider a reversible chemical reaction such as the combination of carbon and oxygen to form carbon dioxide:

$$C(s) + O_2(g) = CO_2(g)$$

At a given temperature and pressure, this reaction will proceed to an equilibrium; that is, the reaction will proceed until a certain ratio of carbon dioxide to carbon and oxygen is attained. After this point is reached no further change will take place unless the reaction conditions are changed. The equilibrium state is defined by the equilibrium constant K which, in the case of the above reaction, is given by equation (1)

$$K = (a_{CO_2})/(a_C)(a_{O_2}) \qquad (1)$$

where a_{CO_2} is the activity of carbon dioxide in the equilibrium mixture, and so on. The activity of a pure solid is one, and the activity of a gas is very nearly equal to its pressure if the total pressure is one atmosphere. Thus, equation (1) simplifies to

$$K = (p_{CO_2})/(p_{O_2}) \qquad (2)$$

under these conditions. It can be seen, therefore, that a knowledge of the equilibrium constant at a given temperature will permit the calculation of the ratio of the pressures of carbon dioxide and oxygen.

The equilibrium constant for a reaction at a given temperature can be computed from the standard free energy change for the reaction at that temperature from equation (3)

$$\Delta F^\circ = -RT \ln K \qquad (3)$$

where R is the gas constant, and T is the absolute temperature. In turn, the standard free energy change can be computed from the standard enthalpy change, ΔH° (the heat of reaction at constant pressure), and the standard entropy change, ΔS°, by equation (4).

$$\Delta F^\circ = \Delta H^\circ - T\Delta S^\circ \qquad (4)$$

All changes in thermodynamic properties are referred to the standard states of the reacting components It is evident, therefore, that the equilibrium constant for a

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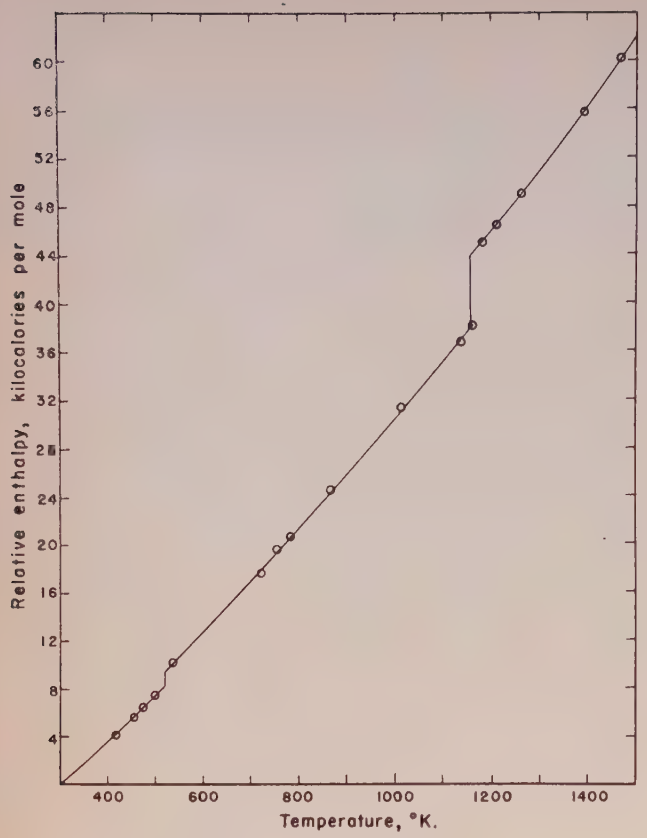

Fig. 1. Variation of the relative enthalpy of sodium sulphate with temperature

reaction can be obtained if the standard enthalpy and entropy changes, or the standard free energy change, are known.

If heat of formation and entropy data are available at some reference temperature for all components of a reaction, and if the enthalpies and entropies of all components at some desired temperature or below the reference temperature are known, then the standard enthalpy and entropy changes at the desired temperature can be calculated by the following equations. In these equations the reference temperature is 298 °K. and the temperature of interest is indicated by T.

$$\Delta H^\circ_{298} = \Sigma(H_{\text{products}})_{298} - \Sigma(H_{\text{reactants}})_{298} \quad (5)$$

$$\Delta S^\circ_{298} = \Sigma(S_{\text{products}})_{298} - \Sigma(S_{\text{reactants}})_{298} \quad (6)$$

$$\Delta H_T^\circ = \Delta H^\circ_{298} + \Sigma(H_T - H_{298})_{\text{products}} - \Sigma(H_T - H_{298})_{\text{reactants}} \quad (7)$$

$$\Delta S_T^\circ = \Delta S^\circ_{298} + \Sigma(S_T - S_{298})_{\text{products}} - \Sigma(S_T - S_{298})_{\text{reactants}} \quad (8)$$

In the system under investigation, many reactions must be considered simultaneously. Equilibrium for the system is attained when the activities of all the components are such as to satisfy the equilibrium constants for all possible reactions. Although the determination of the activities by the simultaneous solution of equilibrium expressions for all possible reactions would be very difficult (if not impossible), a comparison of the equilibrium constants in the various reactions permits one to draw many conclusions regarding the equilibrium of the whole system and, hence, which reactions are most favorable under a given set of conditions.

The elements involved in a sodium-base system are carbon, hydrogen, oxygen, sodium, sulphur, and nitrogen. These elements may be combined in the form of the following basic compounds which might exist, together with the elements themselves, under combustion conditions in a recovery operation: carbon monoxide, carbon dioxide, sulphur monoxide, sulphur dioxide, sulphur trioxide, water, hydrogen sulphide, carbon disulphide, carbonyl sulphide, sodium carbonate, sodium sulphate, sodium sulphide, sodium oxide, and sodium hydroxide. Under the influence of the high temperatures attained in combustion, such compounds as sodium thiosulphate, sodium sulphite, sodium bicarbonate, sodium bisulphate, and sodium bisulphite, if present in the initial spent liquor, would most probably be decomposed to form one or more of the above compounds. Because of this probability, these compounds have been arbitrarily excluded from this study.

The above list of elements and compounds may be considered as comprising the materials actually involved in the reactions occurring during combustion. The reactions leading to the formation of these compounds may be considered as the fundamental reactions involved in combustion since any possible reaction involving these compounds can be obtained by adding or subtracting these formation reactions or multiples thereof. Moreover, it is possible to obtain the equilibrium constant for any reaction involving these compounds, providing the equilibrium constants for the formation reactions are known, by adding or subtracting the logarithms of the necessary equilibrium constants of formation. This, therefore, greatly simplifies the study of the combustion reactions by eliminating the necessity for a direct study of each individual reaction.

The fundamental reactions for the formation of the basic compounds may be written in the following manner:

$$C(s) + O_2(g) = CO_2(g) \quad (1)^*$$

$$C(s) + {}^1/_2O_2(g) = CO(g) \quad (2)$$

$$H_2(g) + {}^1/_2O_2(g) = H_2O(g) \quad (3)$$

$${}^1/_2S_2(g) + O_2(g) = SO_2(g) \quad (4)$$

$${}^1/_2S_2(g) + {}^1/_2O_2(g) = SO(g) \quad (5)$$

$$C(s) + S_2(g) = CS_2(g) \quad (6)$$

$$H_2(g) + {}^1/_2S_2(g) = H_2S(g) \quad (7)$$

$${}^1/_2S_2(g) + {}^3/_2O_2(g) = SO_3(g) \quad (8)$$

$$C(s) + {}^1/_2O_2(g) + {}^1/_2S_2(g) = COS(g) \quad (9)$$

$$Na_2(g) + {}^1/_2S_2(g) = Na_2S(l) \quad (10)$$

$${}^1/_2Na_2(g) + {}^1/_2O_2(g) + {}^1/_2H_2(g) = NaOH(l) \quad (11)$$

$$Na_2(g) + {}^1/_2S_2(g) + 2O_2(g) = Na_2SO_4(l) \quad (12)$$

$$Na_2(g) + C(s) + {}^3/_2O_2\ (g) = Na_2CO_3(l) \quad (13)$$

$$Na_2(g) + {}^1/_2O_2(g) = Na_2O(l) \quad (14)$$

The states of the reactions shown in the above equations were selected solely for convenience in calculation since the states do not influence the end results.

In order to calculate the equilibria in the above basic reactions, it is evident from equations (3) and (8) that heats of formation, entropies, and relative enthalpy and entropy data must be known for all components. Most of the necessary data are available

* In this report the chemical reactions and mathematical equations are numbered separately. When a chemical reaction is referred to by number it will be designated by the word *reaction;* when an equation is referred to by number, it will be designated by the word *equation.*

Table I. Summary of Thermal Data from the Literature

Material	S_{298} Value kg.-cal./-mole deg.	S_{298} Source	H_{298} (form.) Value kg.-cal./-mole	H_{298} (form.) Source	$H_T - H_{298}$ Source	$S_T - S_{298}$ Source
CO_2(g)	51.061	(1)	− 94,051.8	(1)	(2)	(2)
CO(g)	47.301	(1)	− 26,415.7	(1)	(2)	(2)
SO_2(g)	59.40	(1)	− 70,960	(1)	(2)	(2)
SO_3(g)	61.24	(1)	− 94,450	(1)	(2)	(2)
SO(g)	53.04	(1)	8,960	(3)	(2)	(2)
H_2S(g)	49.15	(1)	− 4,815	(1)	(2)	(2)
H_2O(g)	45.106	(1)	− 57,797.9	(1)	(2)	(2)
C(graph.)	1.361	(1)	0	(1)	(2)	(2)
H_2(g)	31.211	(1)	0	(1)	(2)	(2)
O_2(g)	49.003	(1)	0	(1)	(2)	(2)
Na_2(g)	55.02	(1)	33,970	(1)	(2)	(2)
S_2(g)	54.41	(4)	29,860	(1)	(2)	(2)
Na_2S(s)	....	..	− 89,200	(1)	..	..
Na_2O(s)	17.4	(1)	− 99,400	(1)	(2)	(2)
Na_2CO_3(s)	32.5	(1)	−270,300	(1)	..	..
Na_2SO_4(s)	35.73	(1)	−330,900	(1)	..	..
CS_2(g)	56.84	(1)	27,550	(1)	(2)	(2)
COS(g)	55.34	(1)	− 23,800	(1)	(2)	(2)
NaOH(s)	14.2	(4)	−101,990	(1)	(2)	(2)

in the literature (*1–4*); a summary of these data with their sources is shown in Table I.

Since the data for sodium sulphate, sodium carbonate, and sodium sulphide were incomplete, it was necessary to determine the missing values experimentally. Specifically relative enthalpy (the enthalpy at higher temperatures relative to 298°K.) and entropy values were determined for these three compounds; the absolute entropy of sodium sulphide at 25°C. was also determined.

The relative enthalphy measurements were made using a calorimetric technique. In this method the substance under study was dropped from a furnace of known temperature into a calorimeter operating near room temperature. The resulting increase in the calorimeter temperature permitted the calculation of the enthalpy change of the material between the furnace temperature and the final calorimeter temperature. If M represents the moles of the material under study and if $H_{T_2} - H_{T_1}$ is the enthalpy change of the material between T_2 and T_1, then

$$M(H_{T_2} - H_{T_1}) = K_c(T_1 - T_3) \qquad (9)$$

where K_c is the calorimeter constant equal to the mass of the calorimeter multiplied by its heat capacity, T_2 is the temperature of the furnace, T_3 is the initial temperature of the calorimeter, and T_1 is the final temperature of the calorimeter. Since this technique is common practice, the details of the measurements will not be given here.

Obtaining pure anhydrous samples of sodium sulphate and sodium carbonate for these studies presented no problem; however, reasonably pure and anhydrous samples of sodium sulphide were obtained with considerable difficulty. After trying numerous methods in the laboratory, a technique was found which yielded a product of acceptable purity. Briefly, this method was as follows. A sample of sodium sulphide hydrate, ($Na_2S \cdot 9H_2O$) was heated slowly to about 100°C. on an oil bath. At this point the material was molten and was quite fluid. A vacuum was then applied slowly until the water of hydration began to boil; the vacuum was then adjusted to keep the water boiling without severe bumping. When all visible boiling had ceased, the temperature was slowly raised to 140°C., and a slow stream of hydrogen sulphide was passed through

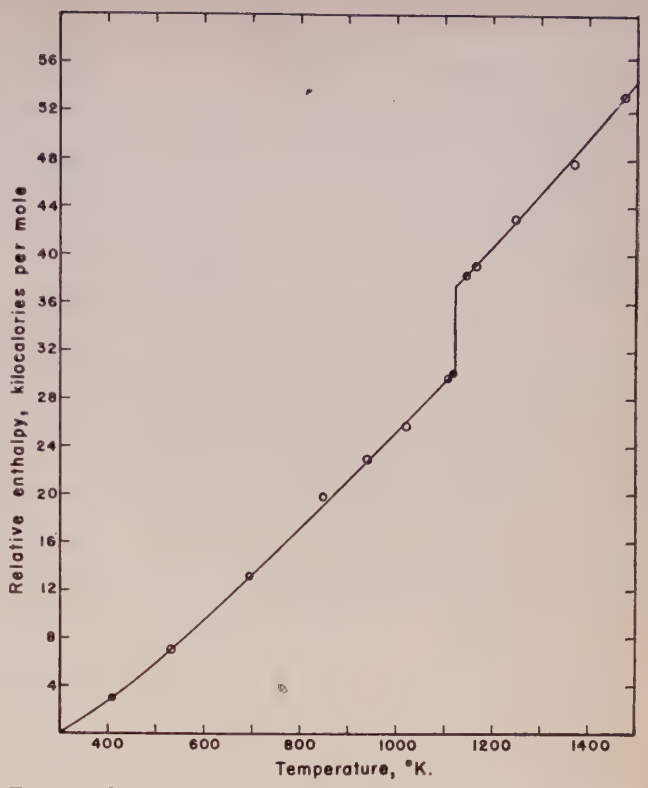

Fig. 2. Variation of the relative enthalpy of sodium carbonate with temperature

the reaction vessel to suppress the hydrolysis of the sodium sulphide. This condition was maintained for a total heating time of 8 hr. The product was then removed and was further dried under vacuum over fresh phosphorus pentoxide for 16 hr. In this manner a product was obtained which analyzed 96.4% sodium sulphide by titration with ammoniacal silver nitrate; this material was used in the enthalpy studies on sodium sulphide.

In making the enthalpy measurements, the thermal decomposition of the sodium carbonate and the oxidation of sodium sulphide were minimized by heating the charges for the shortest possible time in the furnace. Analyses before and after the runs revealed that the changes in composition were negligible.

The results of the relative enthalpy measurements are shown in Figs. 1, 2, and 3. From these curves average values were obtained and are tabulated in Table II. The values given in Table II were used to compute relative entropy values for all three compounds using the relationship

$$(S_T - S_{298}) = \int_{298}^{T} d(H_T - H_{298})/T \qquad (10)$$

$$= (H_T - H_{298})/T + \int_{298}^{T} [(H_T - H_{298})/T^2](dT) \qquad (11)$$

The last integral in equation (11) was solved graphically by plotting $(H_T - H_{298})/T^2$ against T. The relative entropy values so obtained are summarized in Table III. It will be noted that the relative entropy values for sodium sulphide are listed only to 1000°K. This was done because the reliability of the enthalpy measurements on sodium sulphide above this temperature was questionable. Referring to Fig. 3, it can be seen that there was no sharp discontinuity in the enthalpy curve representing the heat of fusion of sodium sulphide;

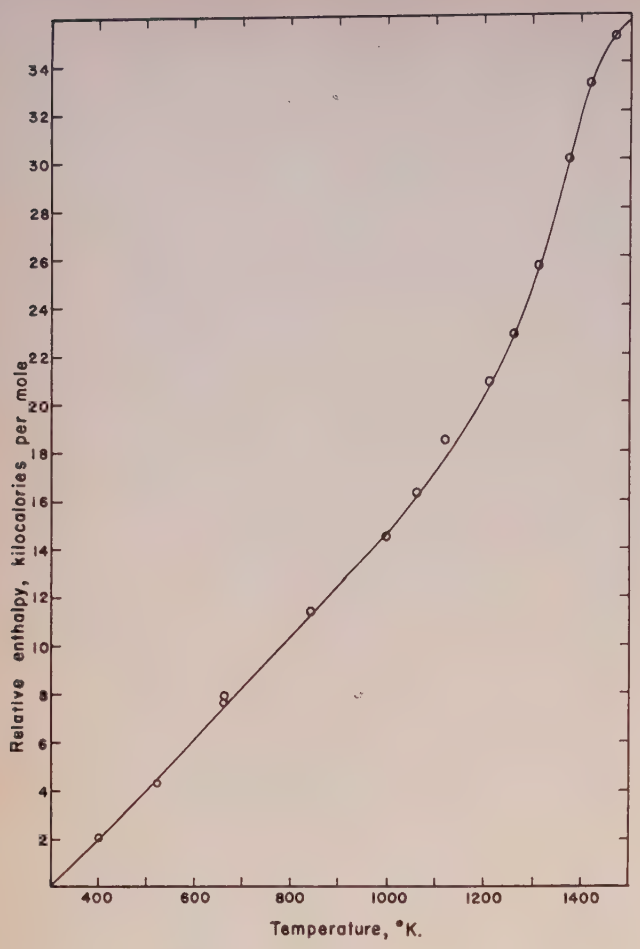

Fig. 3. Variation of the relative enthalpy of sodium sulphide with temperature

instead the curve was S-shaped in the region of fusion (around 1300°K.). It is believed that this phenomenon was the result of impurities in the sodium sulphide sample employed for the measurements, since an impurity having a different fusion temperature would be expected to have a pronounced effect on the measured enthalpy of sodium sulphide near its fusion point. For this reason, only the measured values below the fusion region are considered reliable, and only these values were used in computing the relative entropy of sodium sulphide.

Table II. Relative Enthalpy Values ($H_T - H_{298°K.}$) for Sodium Sulphate, Sodium Carbonate, and Sodium Sulphide

Temperature, °K.	$(H_T - H_{298})$, *Calories per mole* — *Sodium sulphate*	*Sodium carbonate*	*Sodium sulphide*
400	3,650	2,750	2,000
500	7,500	5,900	4,100
520	8,300	6,600	4,500
520	9,450	6,600	4,500
600	12,800	9,500	6,200
700	17,050	13,300	8,300
800	21,350	17,250	10,400
900	25,700	21,250	12,500
1000	30,300	25,300	14,650
1100	35,050	29,350	17,200
1124	36,200	30,400	17,800
1124	36,200	37,500	17,800
1157	38,000	38,850	18,800
1157	43,800	38,850	18,800
1200	45,800	40,800	20,200
1300	50,650	45,300	24,800
1400	56,000	49,800	31,900
1500	61,800	54,500	35,800

Table III. Relative Entropy Values ($S_T - S_{298°K.}$) for Sodium Sulphate, Sodium Carbonate, and Sodium Sulphide

Temperature, °K.	$(S_T - S_{298})$, *Calories per mole* — *Sodium sulphate*	*Sodium carbonate*	*Sodium sulphide*
400	10.5	7.9	5.8
500	19.1	14.9	10.4
520	20.7	16.2	11.2
520	22.9	16.2	11.2
600	28.8	21.5	14.3
700	35.4	27.3	17.5
800	41.1	32.6	20.3
900	46.3	37.3	22.8
1000	51.1	41.6	25.0
1100	55.6	45.4	..
1124	56.6	46.4	..
1124	56.6	52.7	..
1157	58.2	53.9	..
1157	63.2	53.9	..
1200	65.0	55.5	..
1300	68.8	59.1	..
1400	72.8	62.5	..
1500	76.7	65.5	..

The absolute entropy of sodium sulphide at 25°C. was computed from a measurement of the equilibrium in the reaction

$$Na_2SO_4 + 2C = Na_2S + 2CO_2 \qquad (15)$$

Knowing all the necessary thermal data except the entropy of sodium sulphide at 25°C. it was possible to compute this value from the measured equilibrium constant by means of equations (3) through (8).

In conducting this experiment, a static method was used. A Pyrex reactor was charged with anhydrous sodium sulphide and was immersed in a molten lead bath maintained at a constant temperature. The reactor was then evacuated and flushed repeatedly with carbon dioxide in order to replace the air with carbon dioxide. Before proceeding with the determination, the reactor was permitted to stand for 20 hr to attain thermal equilibrium and to permit any gas absorption or desorption to take place. Finally, the pressure in the system was adjusted to 1 atmosphere, the reactor was connected to a manometer, and the measurements were begun. As the reaction proceeded, pressure readings were taken at periodic intervals until no further change in the pressure of the system was detected. This final reading was taken as the equilibrium pressure of carbon dioxide for the reaction. Since the temperatures used were such that the sodium sulphate, carbon, and sodium sulphide were present in the solid form, the equilibrium constant for the reaction was equal to the equilibrium pressure of carbon dioxide.

Two determinations of the equilibrium in reaction (15) were made; the first was conducted at a temperature of 375°C. and led to a value of 18.9 calories/(°K.)(mole) for the entropy of sodium sulphide at 25°C.; the second was conducted at 383°C. and led to a value of 18.3 calories/(°K.)(mole). The average of these two determinations was 18.6 calories/(°K.)(mole) and this was the value used in the subsequent calculations.

Using the thermal data developed in this investigation, together with those available in the literature, it was possible to calculate the equilibria in the basic reactions (1–14). The equilibrium constants so obtained are tabulated as a function of absolute temperature in Table IV. In this table the logarithms of

the equilibrium constants are given for convenience in application.

Having established the equilibria in the basic reactions, it is possible to calculate the equilibrium quite simply for any reaction which can be derived from the basic reactions. This can be best illustrated by a specific example. Consider the reaction

$$2NaOH = Na_2O + H_2O \qquad (16)$$

which is of interest with regard to the combustion of a kraft liquor. This reaction can be derived from the basic reactions by adding reactions (14) and (3) and subtracting reaction (11) multiplied by two. In a similar manner the logarithm of the equilibrium constant for this reaction at any temperature can be obtained by adding the logarithms of the equilibrium constants for reactions (14) and (3) and subtracting twice the logarithm of the equilibrium constant for reaction (11). Thus, for reaction (16) at 600°K., the logarithm of the equilibrium constant can be obtained from the data in Table IV as follows.

$$\log_{10} K = 35.2 + 18.6 - 64.4 = -10.6$$

In addition to reaction (16), a number of other reactions which are considered to be of importance to an understanding of the sodium-base systems have been derived from the basic reactions. These reactions, including reaction (16), are shown below.

$$2NaOH = Na_2O + H_2O \qquad (16)$$

$$Na_2S + 2O_2 = Na_2SO_4 \qquad (17)$$

$$Na_2CO_3 = Na_2O + CO_2 \qquad (18)$$

$$Na_2SO_4 = Na_2O + SO_2 + {}^1/_2O_2 \qquad (19)$$

$$Na_2S + H_2O = Na_2O + H_2S \qquad (20)$$

$$Na_2SO_4 + 2H_2O = 2NaOH + H_2S + 2O_2 \qquad (21)$$

$$Na_2S + {}^3/_2O_2 = Na_2O + SO_2 \qquad (22)$$

$$Na_2SO_4 + C = Na_2O + SO_2 + CO \qquad (23)$$

$$Na_2SO_4 + 2C = Na_2S + 2CO_2 \qquad (24)$$

$$H_2S + {}^3/_2O_2 = H_2O + SO_2 \qquad (25)$$

In addition to the above reactions which are of importance to all three sodium-base processes, several reactions have been considered which are of importance primarily to the neutral or acid sulphite process. These reactions postulate the conversion of sodium sulphide to sodium carbonate.

$${}^3/_4Na_2SO_4 + CO + {}^1/_4Na_2S = Na_2CO_3 + SO \qquad (26)$$

$${}^3/_4Na_2SO_4 + CO_2 + {}^1/_4Na_2S = Na_2CO_3 + SO_2 \qquad (27)$$

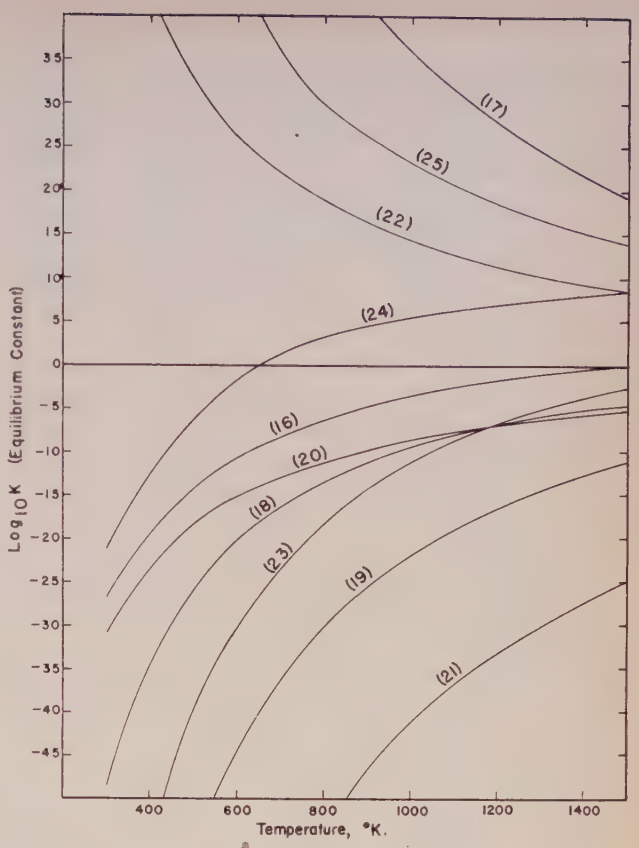

Fig. 4. Equilibrium variation with temperature

(16) $2NaOH = Na_2O + H_2O$
(17) $Na_2S + 2O_2 = Na_2SO_4$
(18) $Na_2CO_3 = Na_2O + CO_2$
(19) $Na_2SO_4 = Na_2O + SO_2 + {}^1/_2O_2$
(20) $Na_2S + H_2O = Na_2O + H_2S$
(21) $Na_2SO_4 + 2H_2O = 2NaOH + H_2S + 2O_2$
(22) $Na_2S + {}^3/_2O_2 = Na_2O + SO_2$
(23) $Na_2SO_4 + C = Na_2O + SO_2 + CO$
(24) $Na_2SO_4 + 2C = Na_2S + 2CO_2$
(25) $H_2S + {}^3/_2O_2 = H_2O + SO_2$

$$Na_2SO_4 + CO = Na_2CO_3 + SO_2 \qquad (28)$$

$$Na_2S + CO_2 + {}^3/_2O_2 = Na_2CO_3 + SO_2 \qquad (29)$$

$$Na_2SO_4 + Na_2S + 2C + 2H_2O = 2Na_2CO_3 + 2H_2S \qquad (30)$$

$${}^1/_4Na_2S + {}^3/_4Na_2SO_4 + C + H_2 = Na_2CO_3 + H_2S \qquad (31)$$

$$2NaOH + Na_2S + 2CO_2 = 2Na_2CO_3 + H_2S \qquad (32)$$

$$Na_2S + CO_2 + H_2O = Na_2CO_3 + H_2S \qquad (33)$$

Still other reactions have been considered which are of interest with regard to the problem, sometimes encountered in kraft recovery operations, of explosions occurring as the smelt drops from the recovery furnace

Table IV. Basic Reaction Equilibria

Reaction	Log_{10} K (*Equilibrium constant*) 298°K.	600°K.	800°K.	1000°K.	1200°K.	1400°K.	1500°K.
(1) $C(s) + O_2(g) = CO_2(g)$	69.09	34.40	25.83	20.68	17.24	14.78	13.80
(2) $C(s) + {}^1/_2O_2(g) = CO(g)$	24.05	14 34	11 93	10 48	9.50	8 79	8.50
(3) $H_2(g) + {}^1/_2O_2(g) = H_2O(g)$	40.05	18.63	13.23	10.06	7.90	6.35	5.73
(4) ${}^1/_2S_2(g) + O_2(g) = SO_2(g)$	59.3	27.5	19.8	15.0	11.8	9.6	8.7
(5) ${}^1/_2S_2(g) + {}^1/_2O_2(g) = SO(g)$	4.66	2 46	1 91	1.58	1.35	1.21	1.14
(6) $C(s) + S_2(g) = CS_2(g)$	1.93	1.11	0.93	0.82	0.75	0.70	0.68
(7) $H_2(g) + {}^1/_2S_2(g) = H_2S(g)$	12.5	5.1	3 18	2.03	1.26	0.72	0.50
(8) ${}^1/_2S_2(g) + {}^3/_2O_2(g) = SO_3(g)$	71.7	31.1	21.3	15.2	11.3	8.4[a]	7.3[a]
(9) $C(s) + {}^1/_2O_2(g) + {}^1/_2S_2(g) = COS(g)$	35.5	17.9	13.6	11.0	9.2	8.0	7.5
(10) $Na_2(g) + {}^1/_2S_2(g) = Na_2S(s,l)$	87.3	36.6	24.3	17.0	11.8[a]	8.3[a]	7.0[a]
(11) ${}^1/_2Na_2(g) + {}^1/_2O_2(g) + {}^1/_2H_2(g) = NaOH(s,l)$	75.5	32 2	21.3	14.9[a]	10.5[a]	7.5[a]	6.3[a]
(12) $Na_2(g) + {}^1/_2S_2(g) + 2O_2(g) = Na_2SO_4(s,l)$	246.7	107.1	73.1	52.8	39.5	30.1	26.4
(13) $Na_2(g) + C(s) + {}^3/_2O_2(g) = Na_2CO_3(s,l)$	201.7	89 7	62 5	46 0	35.5	28 1	25.1
(14) $Na_2(g) + {}^1/_2O_2(g) = Na_2O(s,l)$	84.1	35.2	23.2	16.2	11.3[a]	7.9[a]	6.6[a]

[a] Extrapolated values.

into the dissolving tank. In considering the possibility that these explosions might in part be the result of the formation of elemental sodium in the furnace, equilibria were computed for the following reactions which lead to the formation of elemental sodium under reducing conditions.

$$Na_2S + H_2O + CO = Na_2 + H_2S + CO_2 \quad (34)$$

$$Na_2S + H_2O + C = Na_2 + H_2S + CO \quad (35)$$

$$Na_2CO_3 + CO = Na_2 + 2CO_2 \quad (36)$$

$$Na_2CO_3 + C = Na_2 + CO_2 + CO \quad (37)$$

$$Na_2SO_4 + C = Na_2 + CO_2 + SO_2 \quad (38)$$

$$Na_2O + C = Na_2 + CO \quad (39)$$

$$Na_2O + CO = Na_2 + CO_2 \quad (40)$$

$$2NaOH + C = Na_2 + CO + H_2O \quad (41)$$

$$2NaOH + CO = Na_2 + CO_2 + H_2O \quad (42)$$

The equilibria in reactions (16–25) are shown as a function of temperature in Fig. 4; the equilibria in reactions (26–33) are shown in Fig. 5; and the equilibria in reactions (34–42) are shown in Fig. 6.

The combustion reactions involving carbon, hydrogen, oxygen, and sulphur have been discussed by Whitney, Elias, and May (*5*).

Before analyzing the equilibrium data, a very important point should be kept in mind. Under operating conditions, the gaseous products of the reactions would be swept out of the reaction zone; this would tend to displace the true equilibria in a direction leading to an increase in the amount of the products. The extent of this displacement would depend on the rate of removal of the gases. Thus, in interpreting the reaction equilibria, it should be remembered that the reactions involving gaseous products would give rise to somewhat more of the products under operating conditions than are indicated by the equilibrium data.

Considering first the reactions shown in Fig. 4, several points of general interest to the sodium-base processes are evident. If the combustion is carried out under reducing conditions, then reaction (24), leading to the reduction of sodium sulphate to sodium sulphide, has the most favorable equilibrium of the possible reactions. This is in agreement with the known behavior in the kraft recovery operation. Under these same conditions there are several other points of interest. The equilibria in reactions (16), (20), (18), (23), and (19) reveal that there would be very little sodium oxide formed from reactions involving sodium sulphide, sodium carbonate, sodium sulphate, or sodium hydroxide since the equilibrium constants for all these reactions are quite low even at temperatures up to 1500°K. Moreover, the equilibrium in reaction (21) indicates that the formation of sodium hydroxide from sodium sulphate and water is very unlikely. Considering now combustion under oxidizing conditions, the equilibria in reactions (17), (22), and (25) show that sodium sulphide would be converted primarily into sodium sulphate, although some sodium oxide might also be formed, and that hydrogen sulphide would be converted into sulphur dioxide. The burning of hydrogen sulphide to form sulphur dioxide has an important implication which will be mentioned later.

Consider now the reactions leading to the formation

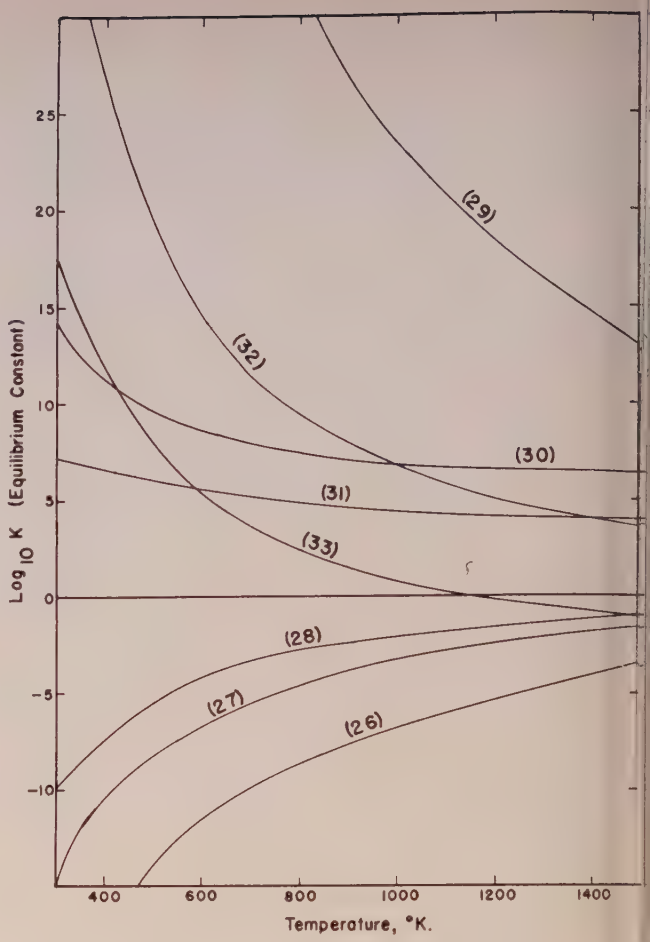

Fig. 5. Equilibrium variation with temperature

(26) $^3/_4Na_2SO_4 + CO + {}^1/_4Na_2S = Na_2CO_3 + SO$
(27) $^3/_4Na_2SO_4 + CO_2 + {}^1/_4Na_2S = Na_2CO_3 + SO_2$
(28) $Na_2SO_4 + CO = Na_2CO_3 + SO_2$
(29) $Na_2S + CO_2 + {}^3/_2O_2 = Na_2CO_3 + SO_2$
(30) $Na_2SO_4 + Na_2S + 2C + 2H_2O = 2Na_2CO_3 + 2H_2S$
(31) $^1/_4Na_2S + {}^3/_4Na_2SO_4 + C + H_2 = Na_2CO_3 + H_2S$
(32) $2NaOH + Na_2S + 2CO_2 = 2Na_2CO_3 + H_2S$
(33) $Na_2S + CO_2 + H_2O = Na_2CO_3 + H_2S$

of sodium carbonate from the sodium-sulphur compounds which are shown in Fig. 5. The equilibria in reactions (26) and (27), involving the production of sodium carbonate from sodium sulphate, sodium sulphide, carbon dioxide, and carbon monoxide, do not appear favorable. Also unfavorable is reaction (28) involving the combination of sodium sulphate and carbon monoxide to give sodium carbonate. The equilibrium in reaction (29) indicates that sodium sulphide would combine with carbon dioxide under oxidizing conditions to give sodium carbonate. However, this reaction would be in competition with the oxidation of sodium sulphide to sodium sulphate, and the latter would probably be the dominant reaction. Reactions (30) and (31), involving the combination of sodium sulphate and sodium sulphide with carbon and water or hydrogen to yield sodium carbonate, also have favorable equilibria; however, under these strongly reducing conditions it is probable that the sodium sulphate present would be completely converted to sodium sulphide before it could combine with sodium sulphide to yield sodium carbonate. Reaction (32), involving the combination of sodium hydroxide with sodium sulphide in the presence of carbon dioxide, also has a favorable equilibrium; however, sodium hydroxide alone will combine with carbon dioxide to yield sodium carbonate

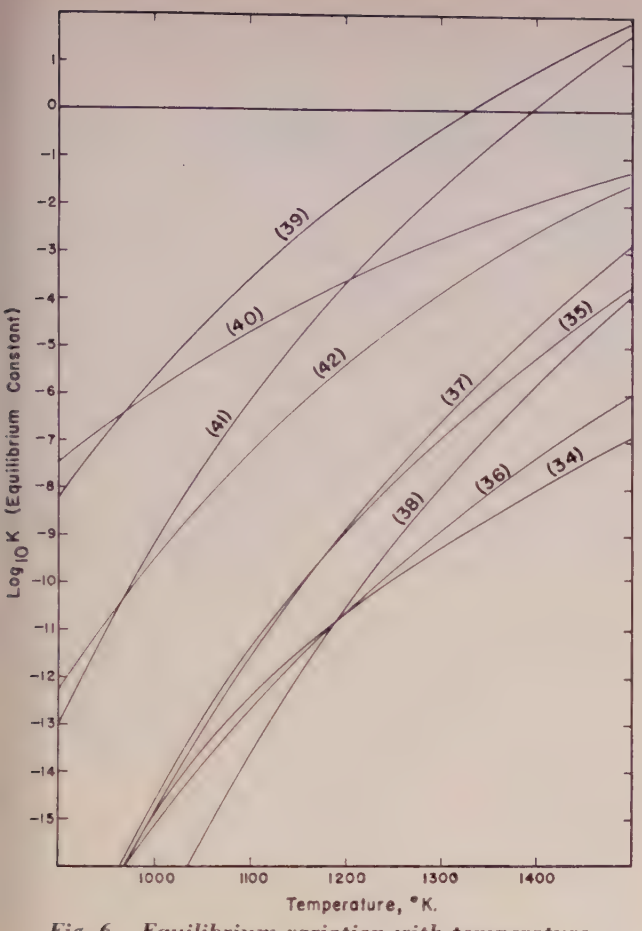

Fig. 6. Equilibrium variation with temperature

(34) $Na_2S + H_2O + CO = Na_2 + H_2S + CO_2$
(35) $Na_2S + H_2O + C = Na_2 + H_2S + CO$
(36) $Na_2CO_3 + CO = Na_2 + 2CO_2$
(37) $Na_2CO_3 + C = Na_2 + CO_2 + CO$
(38) $Na_2SO_4 + C = Na_2 + CO_2 + SO_2$
(39) $Na_2O + C = Na_2 + CO$
(40) $Na_2O + CO = Na_2 + CO_2$
(41) $2NaOH + C = Na_2 + CO + H_2O$
(42) $2NaOH + CO = Na_2 + CO_2 + H_2O$

and this might proceed so rapidly that the reaction involving sodium sulphide would not have time to proceed to equilibrium. The most promising of all the reactions leading to the formation of sodium carbonate appears to be reaction (33). This reaction involves the combination of sodium sulphide with carbon dioxide and water, and the equilibrium is favorable up to a temperature of about 1100°K. However, under operating conditions resulting in the removal of the hydrogen sulphide, the formation of sodium carbonate would be favored at even higher temperatures. This favorable equilibrium should render it possible to convert sodium sulphide to sodium carbonate under the proper operating conditions; this would be most desirable from the standpoint of the recovery of chemicals in the neutral sulphite or sodium-base acid sulphite processes.

At least one way of utilizing this reaction would involve a two-stage operation under controlled conditions. The spent liquor from the sodium-base pulping process might be concentrated and burned under reducing conditions in a combustion chamber such as a conventional kraft process recovery furnace. The resulting smelt, containing sodium sulphide, would then be passed to a reaction chamber consisting of any one of several types of liquid-gas reactors. It might, for example, be a packed tower suitably constructed for intermediate-temperature operation. In this case, the smelt would be treated with carbon dioxide and water vapor to promote the desired reaction. The source of the carbon dioxide and water vapor might be the flue gas from the combustion chamber, the flue gas from a lime kiln, the gas from a carbonating tower, or the flue gas from any other type of furnace employing a fuel of carbonaceous material. The hydrogen sulphide liberated by the reaction would pass out of the reaction chamber with the exit gases and could be burned to yield sulphur dioxide, as shown by reaction (25) which, in turn, could be used in sulphiting a solution of the smelt obtained from the reaction chamber. This would convert the sodium carbonate generated in the reaction chamber to sodium sulphite or sodium bisulphite, which could then be reused in the pulping operation. The carbon dioxide liberated in the sulphiting of the sodium carbonate could be used to enrich the flue gas entering the reaction chamber.

Under conditions providing for the removal of the gaseous products from the reactions shown in Fig. 6 (leading to the formation of elemental sodium), it is conceivable that each of these reactions might proceed sufficiently to liberate some elemental sodium. This is particularly true in the case of sodium oxide and sodium hydroxide. Therefore, it seems quite probable that, under the conditions encountered in kraft furnace operation, some elemental sodium might be formed. This condition would be aggravated by higher than normal temperatures and might explain at least in part, the explosion difficulties sometimes encountered in dissolving the smelt. The solution to this problem would seem to be to operate at the lowest possible furnace temperatures commensurate with the maintenance of the desired reduction reactions which are favored by high temperatures and to avoid overloading the furnace.

There is still another point of interest to be noted from the the reactions leading to the formation of volatile sulphur compounds shown in Figs. 4, 5, and 6. Most of these reactions will proceed appreciably under the conditions usually encountered in kraft furnace operation, and this may in part explain the high sulphur losses relative to the sodium losses which are commonly associated with the kraft recovery process.

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Significance of Laboratory Sizing Data

ELL DEE COMPTON

Laboratory paper sizing data are used to illustrate some of the pitfalls that may be encountered in interpretation of such data. The utility of a simple experimental design and analysis of variance in such work is then illustrated using actual laboratory data.

PAPER research, like most application research, is made interesting, or complex, by the variety of factors which may influence the results of experimental work. The relatively high cost of paper application research is largely a result of the care taken to decrease the influence of undesired variations—experimental errors. At times unrecognized sources of error may influence experimental results so as to lead the chemist to an erroneous conclusion. Statistics is a tool which can be very helpful to the paper chemist in recognizing sources of error, in reaching conclusions with known confidence, and in planning work so as to obtain desired information with minimum effort. The techniques applied here to laboratory data may also be used in plant experimentation.

Ink flotation data from a laboratory application study of four experimental sizing materials are used to illustrate a few potentially useful statistical tools.

It is shown that erroneous conclusions may be drawn from comparison of averages of series of determinations if the amount of overlap in the observations is not taken into consideration.

In this particular study the amount of size applied had an effect on the conclusions drawn from a comparison of sizes. It is demonstrated that the sizes should be compared at a number of levels of size application. A statistical technique is used to estimate quantitatively the importance of the combined effect of size and amount applied.

A study of the sources of experimental error reveals that reproducibility of ink penetration time on the same sheet was much better than the reproducibility from sheet to sheet. It is also found that the position of the sample in the sheet had an effect on the observed time for penetration. Thus it is concluded, based only on this experiment, that one observation per sheet is sufficient, and that the sample should be taken from the center of the sheet.

A re-examination of the data is used to demonstrate that a saving in experimental work may often be realized if the information desired is first stated and then only the work required for reaching these conclusions is carried out.

SOURCE OF DATA

Since it is easier to discuss a specific topic than an abstract subject, a particular experiment was chosen for this illustration of some possible applications of statistics. Four experimental sizing materials were compared in this experiment. It should be emphasized that the conclusions drawn are based *solely* on the data presented and cannot be applied freely to other data nor to other situations. The experiment was performed originally to determine if these four sizing materials differed in application.

In order to have a better basis for comparison, and to minimize the effect of experimental errors, the sizes were investigated at four levels of size application: 0.5, 1.0, 2.0, and 3.0%, based on the dry weight of pulp.

Preparation of Handsheets

Gatineau bleached sulphite pulp (approximately pH 7, before addition of other components) was diluted to 2% consistency with fresh tap water (pH 6.7). The pulp was defibered on a Lightnin Stirrer (20 min.) and refined in a Mead Refiner (1 min.) to a 495 ml. Canadian freeness. The refined pulp was distributed into beakers, 1500 cc., 30 grams dry pulp, per beaker. The required amount of size was added to each beaker and the slurry mixed 20 min. on a laboratory gang stirrer. Thirty-five milliliters of a 3.0% alum solution was added to each beaker and stirred 20 min. The final pH, prior to sheetmaking, was 4.2 to 4.5.

The handsheets were made on a Noble and Wood Laboratory Sheet Machine—forming 8 by 8-in. sheets weighing 2.5 grams.

The order of preparation of the 16 sets (four levels of each of the four sizes) was carefully randomized. A set of four handsheets was made with each size-level treatment. The sheets were numbered consecutively in order of preparation.

The sheets were conditioned 3 days under standard conditions (TAPPI).

Preparation of Test Pieces

A $^3/_4$-in. strip was cut from the left-hand side of the sheet in the direction of the drier movement. A 2-in. strip was then cut and in turn this strip was cut into four 2-in. squares. The piece nearest the operator (edge) was labeled A. The third square was labeled B.

The edges of the squares were folded—$^1/_8$-in. fold—to form the test boats. The boats were formed so that the felt side was placed in the ink.

Ink Flotation Test

The testing was done in a constant temperature-humidity room (TAPPI standard). Carter's blue-black ink adjusted to pH 1.5 with HCl was used. The boats were floated on the ink and the time for penetration recorded to the nearest 5 sec.

The results of the ink flotation tests are shown in Table I with the averages of the eight observations made on each set of sheets.

The average (ink penetration times) of this series of

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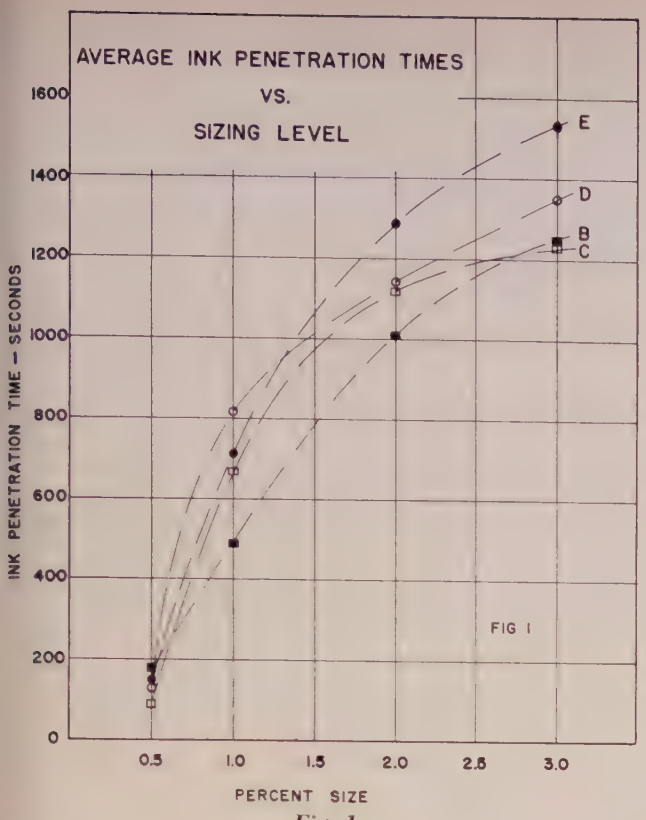

Fig. 1.

size-level data may be connected as shown in Fig. 1 to form fairly smooth "curves."

EXAMINATION OF THE RESULTS

If sound conclusions are to be drawn from the "curves" shown in Fig. 1 it must be known that the points compared at a level (per cent of size applied) are really different before one curve can be judged to be definitely displaced from another. An examination of the points at the 1.0% level of size application may be used to illustrate this point.

The average ink penetration time for sizes B and C are farthest apart. If the individual values of the determinations are plotted as horizontal lines a drawing such as Fig. 2 is obtained. If the upper eight rungs of this ladder were readings obtained on the boats containing size C, and the lower eight on boats containing size B, then it could be said with confidence that on the average it would take longer for the ink to penetrate the boats containing C. Confidence that C was better (higher average ink penetration time) than B would decrease as the rungs from the two sources intermingled as the ink penetration times overlap. If the two sides at the left of Fig. 2 are separated, leaving the rungs from the two sources attached to their respective sides, a picture is obtained of the amount of overlap in the two series of observations, Fig. 2. The amount of overlap is not great, therefore, the conclusion could be drawn that the two sizes really impart different sizing values to the paper.

The method of analysis by ranks (*4*, *5*, *6*) offers a tool for comparison of sets of data such as these so that the probability of making an incorrect decision is known. In comparison of two sets of observations the combined values in the two sets are ranked—assigned numbers in order of increasing magnitude. If more than one observation has the same numerical value the average rank number is assigned to each individual.

If the two samples of data being compared are not significantly different the sum of the rank numbers for each set should be approximately half the total of all the rank numbers. Tables have been prepared (*4*, *6*) in which the probability of chance occurrence of rank totals is given for various numbers of observations. Thus, comparing two sets of observations the lesser rank total is calculated and the probability of obtaining this rank total, or less, may be estimated by entering

Table I. Ink Penetration Time Results in Seconds

Per cent size	*Sheet*	*Boat*	*Size B*	*C*	*D*	*E*
0.5	1	A	200	110	190	160
		B	225	90	110	210
	2	A	210	125	90	150
		B	180	100	110	125
	3	A	95	60	135	125
		B	150	60	160	150
	4	A	160	80	120	170
		B	190	95	150	110
		Av.	176.2	90.0	133.1	150.0
1.0	1	A	580	540	655	610
		B	470	685	695	1020
	2	A	500	540	1000	740
		B	500	570	765	920
	3	A	610	730	655	660
		B	400	900	980	910
	4	A	400	690	750	250
		B	460	680	1045	610
		Av.	490.0	666.9	818.1	715.0
2.0	1	A	960	1235	1025	1290
		B	1075	1020	1400	1510
	2	A	890	1215	1340	1245
		B	1280	1370	955	1080
	3	A	850	1135	1390	1290
		B	950	1350	930	1310
	4	A	990	1260	1010	1220
		B	1080	985	1100	1345
		Av.	1009.4	1196.2	1143.8	1286.2
3.0	1	A	1245	1095	1340	1420
		B	1090	1240	1640	1650
	2	A	1520	1260	1265	1710
		B	1330	1320	1480	1440
	3	A	1205	1210	985	1370
		B	1402	1240	1420	1680
	4	A	970	1150	1010	1350
		B	1195	1260	1645	1640
		Av.	1246.9	1221.9	1348.1	1532.5

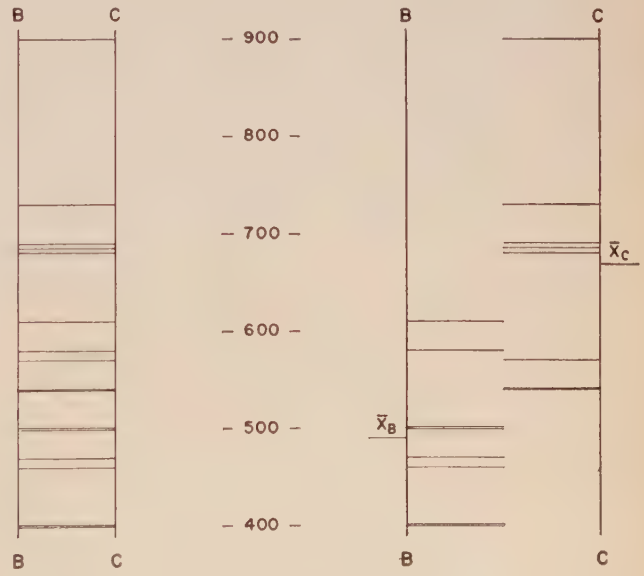

Fig. 2. Comparison of sizes B and C at the 1% level ink penetration times

Table II. Comparison of Sizes B and C at 1% Level Using Ranking of Observations

Observations Arranged for Purposes of Ranking

Size B		Size C	
Ink penetration time, sec.	*Rank*	*Ink penetration time, sec.*	*Rank*
400	1.5	...	..
400	1.5	...	..
460	3	...	..
470	4	...	..
500	5.5	...	..
500	5.5	...	..
...	..	540	7.5
...	..	540	7.5
...	..	570	9
580	10	...	..
610	11	...	..
...	..	680	12
...	..	685	13
...	..	690	14
...	..	730	15
...	..	900	16
	42		94

$P < 0.01$

the proper table. The experimenter must decide if that probability is small enough to warrant calling the two samples different.*

Sizes B and C are compared at the 1% level using analysis by ranks in Table II. In this table the observations are rearranged from their original order so that the assignment of rank numbers is better illustrated. If these sizes were not different each rank total should approximate 68. The sum of the rank numbers for B is 42. Reference to the proper table (*6*) reveals that the probability of obtaining this value by chance, if the sizes were the same, would be less than 1%. Thus it would be concluded that B and C were different at the 1% level of application.

The results obtained with sizes C and D at the 1% level are shown in Fig. 3. Note that the values overlap more than was the case for B and C (Fig. 2). In this

* Throughout this paper it is required that the odds in favor of a correct decision be 20 to 1—a "confidence level" of 95%.

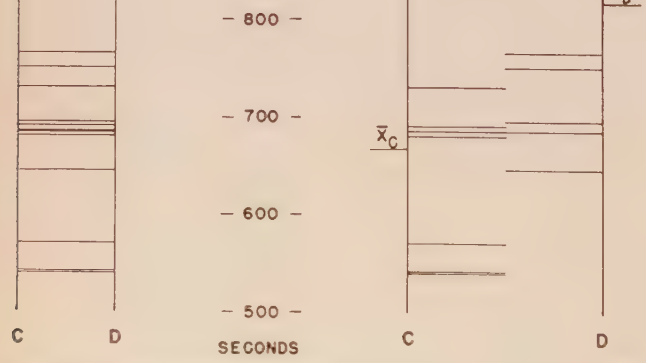

Fig. 3. Comparison of sizes C and D at the 1% level ink penetration times

Table III. Comparison of Sizes C and D at 1% Level Using Ranks

Size C		Size D	
Ink penetration time, sec.	*Rank*	*Ink penetration time, sec.*	*Rank*
540	1.5	655	4.5
685	7	695	9
540	1.5	1000	15
570	3	765	12
730	10	655	4.5
900	13	980	14
690	8	750	11
680	6	1045	16
	50		86

$P > 0.05$

instance it would be more difficult to decide that the two sizes were really different. The comparison by ranks is given in Table III. The decision would be reached that these two sizes did not differ, at the 1% level of application, in their effect on ink penetration time since the lesser rank total (C) could be obtained more than five times in 100 if the sizes were actually the same.

This same technique of comparing two sizes at a given level of application may be used with all of the data. These results are summarized in Table IV.

STATISTICAL CONSIDERATIONS

The procedure, discussed above, of comparing the sizing materials two at a time at each level of application can be a useful tool for the experimenter but it certainly is not the best one for answering the major question: "Are these four sizing materials different?" The tool described above, method of ranks, is one of the simplest and easiest statistical tools but as it was used more work was done than should be necessary in order to answer the primary question. Statistics is most useful only when the information needed is specified—and the proper tool chosen for the job at hand. From here on in this paper an attempt will be made to illustrate this approach; a question will be asked and a statistical tool chosen for answering that question. Alternative routes may be discussed and the one that seems most applicable will be demonstrated.

Are the Four Sizing Materials Different?

Three techniques might be considered:

Comparison of the Slopes and Intercepts of the Ink Penetration—Per Cent Size Curves. This is a quantitative refinement of the general approach frequently employed by the paper chemist. Use of this technique would require that the equation of each curve be known. No simple method (logs, reciprocals, etc.) was found for transforming these curves into straight lines.

Table IV. Conclusions Resulting from Comparison of Sizes Within Levels of Application

Level of application, %	*Conclusions*
0.5	$C < B$; $D < B$; $E \cong B$; $C < E$; $C < E$; $D \cong$
1.0	$B < C$; $B < D$; $B < E$; $C \cong D$; $C \cong E$; $D \cong$
2.0	$B < C$; $B \cong D$; $B < E$; $C \cong D$; $C \cong E$; $D \cong$
3.0	$B \cong C$; $B \cong D$; $B < E$; $C \cong D$; $C < E$; $D \cong$

Definitions: $C < B$ the average ink penetration time for C is *less than* that for B; $D \cong E$ the average ink penetration time for D is not significantly different from that for E.

Further effort did not seem justified in view of other, more easily applied, techniques.

Use of Variance Analysis. This extremely useful and powerful tool has been discussed in such texts as Fisher (*1*), Snedecor (*3*), and Youden (*7*). There is no need to discuss it here. Variance analysis is not applicable in answering the question because two basic assumptions of the technique are not met: the relationship between ink penetration time and size level is not linear and the distribution is not normal. If a simple transformation had been found these transformed data could be used in analysis of variance.

Use of Ranks. The method of ranks is particularly applicable to this problem because no assumptions as to linearity or normality are required. As is the case with all methods using ranked data the method is approximate—it lacks the power of variance analysis—but the speed, simplicity, and freedom from assumptions make it extremely useful. The method described below for comparison of several treatments was developed by Friedman (*2*). It is discussed by Wilcoxon (*6*).

The operations involved in this method of computation are illustrated in Table V, comparing all four of the sizes:

1. Arrange the observations in columns according to the size used such that the observations in each row were taken under similar conditions (size level, sheet number, and boat letter the same in each row).
2. Rank the observations within each *row* (the "r" columns).
3. Add the rank numbers in each column (shown at the bottom of the columns).
4. Square each column rank total.
5. Substitute in the equation:

$$\chi_r^2 = \frac{12}{np(p+1)} \text{(sum of squared column rank totals)} - 3n(p+1)$$

where
n = number of observations per column
p = number of sizes

6. Determine the probability of obtaining the calculated value of χ^2, or larger, by entering a χ^2 table or chart for the proper degrees of freedom $(p-1)$.

The calculation of χ^2 is shown at the bottom of Table V. The test shows that the probability of this combination of rank column totals occurring would be less than one in a thousand if the sizes were the same. Thus, the conclusion may be drawn that the sizes differ.

Which Sizes Differ from Each Other?

Once it has been shown that the sizes do differ, when

Table V. Comparison of Four Sizing Materials

Ink Penetration Time in Seconds

Per cent size	Sheet	Boat	Size B		Size C		Size D		Size E	
			Sec.	r	Sec.	r	Sec.	r	Sec.	r
0.5	1	A	200	4	110	1	190	3	160	2
		B	225	4	90	1	110	2	210	3
	2	A	210	4	125	2	90	1	150	3
		B	180	4	100	1	110	2	125	3
	3	A	95	2	60	1	135	4	125	3
		B	150	2.5	60	1	160	4	150	2.5
	4	A	160	3	80	1	120	2	170	4
		B	190	4	95	1	150	3	110	2
1.0	1	A	580	2	540	1	655	4	610	3
		B	470	1	685	2	695	3	1020	4
	2	A	500	1	540	2	1000	4	740	3
		B	500	1	570	2	765	3	920	4
	3	A	610	1	730	4	655	2	660	3
		B	400	1	900	2	980	4	910	3
	4	A	400	2	690	3	750	4	250	1
		B	460	1	680	3	1045	4	610	2
2.0	1	A	960	1	1235	3	1025	2	1290	4
		B	1075	2	1020	1	1400	3	1510	4
	2	A	890	1	1215	2	1340	4	1245	3
		B	1280	3	1370	4	955	1	1080	2
	3	A	850	1	1135	2	1390	4	1290	3
		B	950	2	1350	4	930	1	1310	3
	4	A	990	1	1260	4	1010	2	1220	3
		B	1080	2	985	1	1100	3	1345	4
3.0	1	A	1245	2	1095	1	1340	3	1420	4
		B	1090	1	1240	2	1640	3	1650	4
	2	A	1520	3	1260	1	1265	2	1710	4
		B	1330	2	1320	1	1480	4	1440	3
	3	A	1205	2	1210	3	985	1	1370	4
		B	1420	2.5	1240	1	1420	2.5	1680	4
	4	A	970	1	1150	3	1010	2	1350	4
		B	1195	1	1260	2	1645	4	1640	3
	Σr			65.0		63.0		90.5		101.5
	$(\Sigma r)^2$			4225.0		3969.0		8190.25		10,302.25

$$\chi_r = \frac{12}{np(p+1)} \text{(sum of squared column rank totals)} - 3n(p+1)$$

$$= \frac{12}{32 \times 4(5)} (4225.0 + 3969.0 + 8190.25 + 10,302.25) - 3 \times 32 (4 \times 1) = \frac{12}{640} (26,686.5) - 480$$

$$\chi_r^2 = 20.4$$

$$P < 0.001$$

considered as a group, the next question might well be: which sizes differ from each other? A method using ranked data once again seems most applicable—essentially because of the freedom from the linearity and normality assumptions.

The technique chosen for answering this question was developed by Wilcoxon (*4*, *6*). Unlike Friedman's χ_r^2 test it can be applied to only two (materials) treatments where the materials are evaluated under more than one set of conditions. Wilcoxon's method was chosen in preference to Friedman's because the χ_r^2 test tends to give somewhat more conservative answers, where only two or three treatments are used, than the tabulated probabilities would indicate (*2*). Thus, in a borderline decision there would be some chance of deciding that the materials did not differ significantly, using the χ^2 test, where the 95% confidence requirement had actually been met.

This tool of Wilcoxon's is well illustrated in answering the second question. The operations may be itemized:

1. At each level of size application rank the observations made on *both* sizes being compared exactly as it was previously done.
2. Sum the rank numbers for each size at each level.
3. Sum the totals of rank numbers for each size (obtained in 2).
4. Enter the proper table (Wilcoxon (*6*)) to determine the probability of obtaining the rank totals computed in 3.

The application of this tool for comparing two materials is illustrated in Table VI by comparing size C to size D.

Since the grand total of rank numbers assigned to size C would be expected to occur less than 1 time in 100 if C and D were the same, C could be judged a less effective size than D when considered at the four levels of application studied.

This same test can be applied to the other five combinations of the sizes with these conclusions:

Size B not different from size C
Size B not different from size D
Size B different from size E
Size C different from size E
Size D different from size E

(It may be noted that these conclusions do not agree exactly with each of the statements made when the sizes were compared at each level. These conclusions, however, are broader since all of the information—all levels—was used in the calculations.)

Table VI. Comparison of Size C to Size D

Ink Penetration Time in Seconds

Per cent size	Sheet	Boat	Size C — Sec.	Size C — r	Size D — Sec.	Size D — r
0.5	1	A	110	9	190	16
		B	90	4.5	110	9
	2	A	125	12	90	4.5
		B	100	7	110	9
	3	A	60	1.5	135	13
		B	60	1.5	160	15
	4	A	80	3	120	11
		B	95	6	150	14
				44.5		91.5
1.0	1	A	540	1.5	655	4.5
		B	685	7	695	9
	2	A	540	1.5	1000	15
		B	570	3	765	12
	3	A	730	10	655	4.5
		B	900	13	980	14
	4	A	690	8	750	11
		B	680	6	1045	16
				50		86
2.0	1	A	1235	10	1025	6
		B	1020	5	1400	16
	2	A	1215	9	1340	12
		B	1370	14	955	2
	3	A	1135	8	1390	15
		B	1350	13	980	1
	4	A	1260	11	1010	4
		B	985	3	1100	7
				73		63
3.0	1	A	1095	3	1340	12
		B	1240	6.5	1640	15
	2	A	1260	8.5	1265	11
		B	1320	10	1480	14
	3	A	1210	5	985	1
		B	1240	6.5	1420	13
	4	A	1150	4	1010	2
		B	1260	8.5	1645	16
				52		84
			Totals	219.5		324.5

$P < 0.01$

Does the Level at Which the Sizes Are Compared Affect the Conclusion?

The original question, the purpose of the preparation of the sizes, has been answered. Other information, however, may be obtained from the data. It is possible that this additional information might be as useful as that already obtained.

From an inspection of Fig. 1 it would seem that these four sizes behave differently as the amount applied is changed—i.e., the shapes of the curves (as estimated from the data and shown in Fig. 1) are quite different. The various tests of size against size would also lend support to this belief. The importance of the effect of the amount of size applied on any conclusion reached in a comparison of the materials may be estimated by a statistical technique. This joint effect of size and level, termed *interaction* by the statistician, is usually investigated in variance analysis. Variance analysis cannot be used in this problem because of failure of the underlying assumptions.

Wilcoxon (*6*) has extended Friedman's χ_r^2 technique to include a test of significance of interactions. This test is slightly more complicated than those previously described but is still less involved than variance analysis. The following steps are followed in the computation (these steps are given specifically for the example in order to make it as clear as possible):

1. Using measurements for corresponding sheets and boats, and each size, tabulate the difference between levels 3.0 and 2.0.
2. Rank these differences, among the sizes (same sheet and boat numbers) assigning the lowest rank number to the greatest negative difference (if any).
3. Sum these rank numbers (2).
4. Using these rank totals (3) calculate:

$$\chi^2_{rL_3-L_2} = \frac{12}{np(p+1)} (\text{sum of squared column (size) rank total}) - 3n(p+1)$$

5. Using measurements for corresponding sheets and boats, and each size, tabulate the *sum* of levels 3.0 and 2.0 *minus* 2 times the 1.0 level.
6. Rank these data (5) as in (2).
7. Sum these rank numbers (6).
8. Using these rank totals (3) calculate: $\chi^2_{rL_3+L_2-L_1}$.
9. Using measurements for corresponding sheets and boats, and each size, tabulate the *sum* of levels 3.0, 2.0, and 1.0 minus 3 times the (5) level.

Table VII. A Test of the Significance of the Size-Level Interaction

Data Entered Are the Indicated Differences of Ink Penetration Times (Sec.) for the Stated Levels of Size Application

Sheet	Boat	Size B		Size C		Size D		Size E	
		Sec.	r	Sec.	r	Sec.	r	Sec.	r
				1. $L3.0 - L2.0$					
1	A	285	3	−140	1	315	4	130	2
	B	15	1	220	3	240	4	140	2
2	A	630	4	45	1	75	2	465	3
	B	50	2	−50	1	525	4	360	3
3	A	400	4	75	2	−405	1	80	3
	B	470	3	−110	1	490	4	450	2
4	A	−20	2	−110	1	0	3	130	4
	B	115	1	275	2	545	4	295	3
	Σr		20		12		26		22
	$(\Sigma r)^2$		400		144		676		484
		2. $L3 + L2 - 2 \times L1$							
1	A	1045	1	1250	3	1055	2	1490	4
	B	1225	3	890	1	1650	4	1120	2
2	A	1410	3	1395	2	605	1	1475	4
	B	1610	4	1550	3	905	2	680	1
3	A	835	1	885	2	1065	3	1340	4
	B	1570	4	790	2	390	1	1170	3
4	A	1160	3	1030	2	520	1	2070	4
	B	895	3	885	2	655	1	1765	4
	Σr		22		17		15		26
	$(\Sigma r)^2$		484		289		225		676
		3. $L_3 + L_2 + L_1 - 3 \times L_{0.5}$							
1	A	2185	1	2540	3	2450	2	2840	4
	B	1960	1	2675	2	3405	3	3550	4
2	A	2280	1	2640	2	3335	4	3245	3
	B	2570	1	2960	3	2870	2	3065	4
3	A	2380	1	2895	3	2625	2	2945	4
	B	2320	1	3310	3	2850	2	3450	4
4	A	1880	1	2860	4	2410	3	2310	2
	B	2165	1	2640	2	3340	4	3265	3
	Σr		8		22		22		28
	$(\Sigma r)^2$		64		484		484		784

$$\chi^2_{r_{L_3-L_2}} = \frac{12}{np(p+1)} \text{ (sum of squared column rank totals)} - 3n(p+1)$$

$$= \frac{12}{8 \times 4(4+1)} (400 + 144 + 676 + 484) - 3 \times 8(4+1)$$

$$= \frac{3}{40} (1704) - 120$$

$$\chi^2_{r_{L_3-L_2}} = 7.8$$

$$\chi^2_{r_{L_3+L_2-2\times L_1}} = \frac{12}{np(p+1)} \text{ (sum of squared column totals)} - 3n(p+1)$$

$$= \frac{12}{8 \times 4(4+1)} (484 + 289 + 225 + 676) - 3 \times 8(4+1)$$

$$= \frac{3}{40} (1674) - 120$$

$$\chi^2_{r_{L_3+L_2-2\times L_1}} = 5.5$$

$$\chi^2_{r_{L_3+L_2+L_1-3L_{0.5}}} = \frac{12}{np(p+1)} \text{ (sum of squared column totals)} - 3n(p+1)$$

$$= \frac{12}{8 \times 4(4+1)} (64 + 484 + 484 + 784) - 3 \times 8(4+1)$$

$$= \frac{3}{40} (1816) - 120$$

$$\chi^2_{r_{L_3+L_2+L_1-3L_{0.5}}} = 16.2$$

4. Calculation of $\chi^2_{r\text{interaction}}$

$$\chi^2_{r\text{interaction}} = \chi^2_{r_{L_3-L_2}} + \chi^2_{r_{L_3+L_2-2L_1}} + \chi^2_{r_{L_3+L_2+L_1-3L_{0.5}}}$$

$$= 7.8 + 5.5 + 16.2$$

$$\chi^2_{r\text{interaction}} = 29.5$$

Degrees of freedom $= 3 + 3 + 3 = 9$

$P < 0.001$

10. Rank these data (9) as in (2) and (6).
11. Sum these rank numbers (10).
12. Using these rank totals (11) calculate: $\chi^2_{rL_3+L_2+L_1-3\times L_{0.5}}$.
13. Sum the three χ^2_r's:

$$\chi^2_{r\,L_3-L_2} + \chi^2_{r\,L_3+L_2-L_1} + \chi^2_{r\,L_3+L_2+L_1-3\times L_{0.5}} = \chi^2_{r\,\text{interaction}}$$

14. Determine, from a chart or table, the probability of obtaining this value of χ^2 for the proper degrees of freedom.

$$(p_{L_3-L_2} + p_{L_3+L_2-2L_1} + p_{L_3+L_2+L_1-3L_{0.5}})$$

The application of Wilcoxon's method of testing the importance of an interaction is illustrated in Table VII. Steps 1 to 4 are included in part 1 of the table, steps 5 to 8 in part 2, steps 9 to 12 in part 3, and step 13 in part 4.

The probability of obtaining the χ^2 calculated for the interaction, if the interaction was unimportant, would be less than one in 1000. Thus, the chemist could conclude that the interaction is very important.

This calculation has, in essence, given a quantitative measure of the validity of the possible conclusion that might have been drawn from an inspection of Fig. 1—that the curves differ as much in shape as they do in position.

This conclusion might be extended to an inference that these four sizes differ in mode of action as well as in the amount of sizing imparted to the paper.

Is the Observed Ink Penetration Time Dependent on the Sequence of Sheetmaking?

The ink penetration time results vary rather widely among the eight observations made on each size level. For purposes of planning future work it might be profitable to investigate the reasons for this variation—within the limits of the data.

One effect that might be investigated is the possibility of a trend in ink penetration results with the order of sheet preparation, or, as phrased above, "Is the observed ink penetration time dependent on the sequence of sheetmaking?" The χ^2_r tool that was used in comparing the sizes may be used.

In this illustration of the χ^2_r tool the columns are made up of sheets of the same order number—the first sheet made, second sheet, etc. Each row then is formed of observations made on these sheets where the size, size level, and boat were common to all column values.

Table VIII. A Test of the Influence of Order of Sheet Preparation on Ink Penetration Time

Level	Size	Boat	Sheet 1		Sheet 2		Sheet 3		Sheet 4	
			Sec.	r	Sec.	r	Sec.	r	Sec.	r
0.5	B	A	200	3	210	4	95	1	160	2
		B	225	4	180	2	150	1	190	3
	C	A	110	3	125	4	60	1	80	2
		B	190	2	100	4	60	1	95	3
	D	A	190	4	90	1	135	3	120	2
		B	110	1.5	110	1.5	160	4	150	3
	E	A	160	3	150	2	125	1	170	4
		B	210	4	125	2	150	3	110	1
1.0	B	A	580	3	500	2	610	4	400	1
		B	470	3	500	4	400	1	460	2
	C	A	540	1.5	540	1.5	730	4	690	3
		B	685	3	570	1	900	4	680	2
	D	A	655	1.5	1000	4	655	1.5	750	2
		B	695	1	765	2	980	3	1045	4
	E	A	610	2	740	4	660	3	250	1
		B	1020	4	920	3	910	2	610	1
2.0	B	A	960	3	890	2	850	1	990	4
		B	1075	2	1280	4	950	1	1080	3
	C	A	1235	3	1215	2	1135	1	1260	4
		B	1020	2	1370	4	1350	3	985	1
	D	A	1025	2	1340	3	1390	4	1010	1
		B	1400	4	955	2	930	1	1100	3
	E	A	1290	3.5	1245	2	1290	3.5	1220	1
		B	1510	4	1080	1	1310	2	1345	3
3.0	B	A	1245	3	1520	4	1205	2	970	1
		B	1090	1	1330	3	1420	4	1195	2
	C	A	1095	1	1260	4	1210	3	1150	2
		B	1240	1.5	1320	4	1240	1.5	1260	3
	D	A	1340	4	1265	3	985	1	1010	2
		B	1640	3	1480	2	1420	1	1645	4
	E	A	1420	3	1710	4	1370	2	1350	1
		B	1650	3	1440	1	1680	4	1640	2
		Σr		86.5		87.0		72.5		74.0
		$(\Sigma r)^2$		7482.25		7569		5256.25		5476

$$\chi^2_r = \frac{12}{np(p+1)} \text{ (sum of squared column rank totals)} - 3n(p+1)$$

$$= \frac{12}{32\times4(4+1)} (7482.25+7569+5256.25+5476) - 3\times32\,(4+1)$$

$$= \frac{12}{640} (25{,}783.5) - 480$$

$$\chi^2_r = 3.44$$

$$P \cong 0.34$$

Since the observed value of χ^2 would be expected to occur as often as 34 times in 100 by chance, if the sheets were not different, it would be logical to conclude that the order of sheetmaking had no significant effect on the observed ink penetration time.

Does the Observed Ink Penetration Time Depend on the Location of the Test Boat in the Sheet?

It is quite possible that the ink penetration time observed on any boat might be influenced by nonuniformity of the sheet. Some measure of sheet uniformity is available from the data since care was taken to cut the boats from previously selected areas of each sheet.

Thus, if the A boats should give results significantly different from those obtained with B boats the chemist would be confident that the sizing characteristics differed at the two areas chosen. The observer also introduces a source of variation but his error would affect all the observations equally since the order of determination was carefully randomized.

Another type of analysis by ranks developed by Wilcoxon (*4, 6*) is particularly applicable in this problem. The test is for "paired replicates." The steps in the computation are listed:

1. Tabulate the algebraic difference between the members of each pair.
2. Rank the differences—ignoring the sign.
3. Give the rank number the sign of the corresponding difference.
4. Total the rank numbers of each sign.
5. Enter the proper table to determine the probability of a chance occurrence of these rank totals.

This method of studying paired replicates by ranked data is illustrated in Table IX. The observed data are not tabulated (cf. Table I), only the differences and ranks are given.

Since the rank totals obtained would occur by chance only about 16 times in 1000 (if the results found on the respective boats were not different) a statement that the position of the test boat has an effect on the observed ink penetration time may be made with considerable confidence.

Which Causes the Greater Variation in Observed Ink Penetration Time, Differences from Sheet to Sheet or Nonuniformity Within Sheets?

The relatively large number of observations required for valid conclusions in work such as described here is a result of the broad spread of results obtained under seemingly identical conditions. If this spread of the results could be decreased it would be possible to reduce the amount of testing—with equally valid conclusions. Thus, if the major source, or sources, of error can be determined it would be possible to reduce the amount of work in future experiments by concentrating the effort on the sources of greatest variation—or perhaps to make improvements in technique. The data presented in this paper permit a comparison of the experimental errors that originate in nonreproducibility of sheets with those due to nonuniformity within sheets.

The variance of a sample of observations is often used as a measure of the spread of the observations. It is calculated by squaring the difference of each observation from the mean, adding these squares, and dividing the sum by the number of observations less one. The equation is:

$$\text{variance} \equiv S^2 = \frac{\Sigma(x - \bar{x})^2}{n - 1}$$

where

x = an observation
$\bar{x}$ = the mean of the observations
n = the number of observations
Σ = summation

This equation may be rearranged to a form that is generally more useful for calculation:

$$S^2 = \frac{\Sigma x^2 - (\Sigma x)^2/n}{n - 1}$$

The spreads of two samples of observations may be compared by comparing the variances of these samples. Fisher (*1*) developed a test for comparison of variances which Snedecor (*3*) named the *F ratio* in his honor. In order to compare two variances the chemist divides the larger by the smaller and refers to a table of F ratios, using the proper degrees of freedom (one less than the number of observations used in computing the variance) for each variance to determine if the F obtained might have occurred by chance. This statistical tool is known as *analysis of variance*. Analysis of variance was not applicable in the previous investigations due to the failure of the linearity and normality assumptions. In this comparison of two sources of experimental error (among different sheets versus within sheets) these assumptions appear to be valid since the nonlinearity observed with the change of level of size application does not affect the calculations—the calculations are dependent on the variations in ink penetration times among sheets made at a size level and the variation in results obtained with the two boats from a sheet.

The variance arising from a common source but under different conditions (as with a different size at a different level) in an experiment may be added. Thus, in this example the variance among sheets at each of the 16 size-level treatments may be calculated and the 16 variances added to give the best picture of the spread of the observations due to variations among sheets.

The variance at a size level would be calculated by the equation:

$$S^2_{A_{S-L}} = \frac{\Sigma x^2_{S-L} - (\Sigma x_{S-L})^2/4}{4 - 1}$$

where

$S^2_{A_{S-L}}$ = variance among sheets at a size level
x_{S-L} = an observation made at a size level
4 = the number of sheets used at a size level

The total variance among sheets in the experiment would be:

$$S^2_A = \frac{\Sigma[\Sigma x^2_{S-L} - (\Sigma x_{S-L})^2/4]}{16[4-1]}$$

Substituting the observed data (Table I) in the equations yields the following value:

$$S^2_A = 47{,}755$$

The variance due to the position of the boat used (A or B) within the sheet may be calculated in the same

Table IX. Effect of Boat Position on Observed Ink Penetration Time

Data Shown Are Differences in Observed Value for Boat A Minus Observed Value for Boat B

Level	Sheet	Size B		Size C		Size D		Size E	
		Diff.	r	Diff.	r	Diff.	r	Diff.	r
0.5	1	−25	−10	20	6	80	24	−50	−20
	2	30	15	25	10	−20	−6	25	10
	3	−45	−19	0	1.5	−25	−10	−25	−10
	4	−30	−15	−15	−4	−30	−15	60	22
1.0	1	110	28.5	−145	−32.5	−40	−18	−410	−61
	2	0	1.5	−30	−15	235	48	−180	−38
	3	210	40	−170	−37	−325	−56	−250	−49
	4	−60	−22	10	3	−295	−53	−360	−57
2.0	1	−115	−30	215	42.5	−375	−58	−220	−45
	2	−390	−60	−155	−34.5	385	59	165	36
	3	−110	−27	−215	−42.5	460	63	−20	−6
	4	−90	−25.5	275	51	−90	−25.5	−125	−31
3.0	1	155	34.5	−145	−32.5	−300	−54	−230	−47
	2	190	39	−60	−22	−215	−42.5	270	50
	3	−215	−42.5	−30	−15	−435	−62	−310	−55
	4	−225	−46	−110	−28.5	−635	−64	−290	−52
	$\Sigma + r$		158.5		114.0		194.0		118.0

Sum of all + rank numbers = 584.5

$P \cong 0.016$

manner except that the first equation given for the variance is most useful. The equation would be:

For the variance between boats within a sheet:

$$S^2_{B_W} = \frac{\Sigma(x - \bar{x})^2}{2-1}$$

For all boats:

$$S^2_B = \frac{\Sigma[\Sigma(x-x)^2]}{64[2 - 1]}$$

Substitution of the observed data in the equations yields the following value:

$$S^2_B = 22{,}919$$

The variance among sheets (S^2_A) appears to be greater than the variance within sheets (S^2_B). This can be tested using the F ratio:

$$F = \frac{S^2_A}{S^2_B} = \frac{47{,}755}{22{,}919} = 2.08$$

Referance to a table of F ratios (*1*, *3*, *7*), using the proper degrees of freedom (the denominators in the variance calculations) shows that an F of 2.08, for 48 and 64 degrees of freedom, would occur by chance less than one time in 100 if the variances were the same. Thus, it may be judged that the variation among sheets was actually greater than that within sheets.

With this information available a decision might be made to try to improve the reproducibility from sheet to sheet and thus sharpen the ink penetration test as a tool for comparing sizing materials. If there is little possibility of improvement in sheet-to-sheet uniformity it would be best to concentrate future testing on more sheets rather than on replicate observations on sheets, since less information is obtained by checking the observation on a sheet than is obtained by running more sheets.

Could the Sizes Have Been Shown to Be Different with Fewer Observations?

After examination of the data accumulated in the previous section it would seem that it might have been possible to show that the sizes were different with fewer observations than were actually made. Certainly the confidence in the statement that the sizes were different (6.13) was greater—99.9% $(1-P)$—than the requirement originally set—95%. As a check on the possibility that fewer observations would have been sufficient to show the sizes different, the data may be reexamined.

Since it was shown that there was a greater variation among sheets than within sheets, one observation on each sheet might be enough. It is possible that three sheets would be sufficient. For this investigation the ink penetration times observed with the B boats on sheets 1, 2, and 3 will be used. The χ^2_r test employed in the previous study is used. Actually there is no need to rank the observations again—the rank numbers assigned to the B boats for sheets 1, 2, and 3 in Table V may be accumulated and the χ^2_r calculated:

$$\chi^2_r = \frac{12}{12 \times 4(4+1)}(676 + 484 + 1056.25 + 1560.25) - 3 \times 12(4+1)$$

$$= \frac{3776.5}{20} - 180$$

$$\chi^2_r = 8.82$$

$$P = 0.033$$

This value of χ^2_r could be expected to occur by chance only 33 times in 1000. There the conclusion may be drawn that the size materials are different. This is the same conclusion as previously reached, with less confidence it is true, but with more confidence than was originally required for conclusions—and with less than half the observations.

Although it may not be possible to extend directly the results of this study to future experiments, the information accumulated here could certainly be used as a basis for planning future work. If the experimenter believes that the errors in this work were typical of what he can expect, he could certainly reduce the amount of testing when he wants to compare experimental sizes again. If any new sizes to be compared differ as much as these did, and the errors are comparable, then the number of observations considered

in this last section would be adequate to determine if the sizes differ—if this is the only information desired.

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The Determination of Copper in Wood Pulps with Tetraethylenepentamine

R. M. KINGSBURY and C. L. LAKE

A simple and rapid method is described for colorimetric determination of copper in cellulosic material. The sample of material is digested with nitric, sulphuric, and perchloric acids, is neutralized and filtered if necessary, and a blue complex is developed with tetraethylenepentamine. The amount of copper is determined by comparing the transmittancy of the solution to that of standard copper solutions by using a photometer with a filter having maximum transmission at 595 to 640 mmu. The sensitivity, when a 10-gram sample is used, is about 1 p.p.m. as copper.

Although there are numerous reagents for the colorimetric determination of copper, the most sensitive ones (*5*) require a time-consuming separation of the copper from interfering substances by precipitation or extraction with organic solvents. A simpler and more rapid procedure based on the use of tetraethylenepentamine as suggested by Crumpler (*1*) has been developed at the Forest Products Laboratory for determining copper in cellulosic materials. In this procedure the cellulosic material is dissolved by wet-ashing with nitric, sulphuric, and perchloric acids, and the tetraethylenepentamine is added after carefully neutralizing and filtering the solution. The concentration of copper in the sample is obtained by comparing the intensity of color in the solution with that of standard copper solutions treated by the same procedure.

Extinction data reported by Crumpler and results obtained at the Forest Products Laboratory indicate that the color intensity of the copper-amine complex is sufficient to provide a sensitivity of about 1 p.p.m. if a sample weight of 10 grams of material is used.

DESCRIPTION OF THE METHOD

Special apparatus consists of:

A glass aspirator pump and adapter tube (*2*).

A spectrophotometer or filter photometer with monochromatic filter having maximum transmission at 595 to 640 mmu and an absorption cell with a light path of at least 50 mm.

The reagents required are:

Concentrated sulphuric acid, C.P. reagent grade.

Concentrated nitric acid, C.P. reagent grade.

Perchloric acid, 70 to 72% ACS standard

Phosphoric acid, 75%, C.P. reagent grade.

Tetraethylenepentamine, technical, Carbide and Carbon Chemical Corp., 2% aqueous solution.

Ammonium hydroxide, C.P. reagent diluted 1 to 1.

Red litmus paper.

Standard copper solution prepared as follows: Dissolve 1.964 grams of pure uneffloresced cupric sulphate in distilled water, add enough hydrochloride or sulphuric acid to make the final acidity 0.1 normal and dilute to 1 liter. This solution contains 0.5 gram of copper per liter, and a weaker solution (conveniently 0.1 gram of copper per liter) should be made for preparation of the standards for color comparison by diluting a portion of the strong solution.

The standard copper solution may also be made by dissolving pure copper in 1:1 nitric acid and boiling to expel oxides of nitrogen before diluting to volume.

Prepare a representative sample by cutting or tearing the pulp into pieces about $^1/_2$ in. square if lapped or sheeted. Wood samples should be in the form of shavings or ground to pass a 40-mesh screen. For convenience in weighing, the sample should be air-dry and have a known moisture content to permit weighing an amount equivalent to a definite weight of moisture-free material. Samples must be handled with great care to avoid contamination by soiling, grinding, or exposure to dust.

Procedure

Rinse all glassware with hot dilute hydrochloric acid and distilled water before using.

Weigh out an amount of air-dry material equivalent to 10.0 grams of moisture-free material and place it in a 1-liter Kjeldahl flask. Add 60 ml. of concentrated nitric acid and 10 ml. of concentrated sulphuric acid and heat gently over a low flame while whirling the flask until a vigorous reaction with evolution of nitrous oxide starts. Remove the flask from the flame and place it on a stand under a fume hood. It is advisable to draw off the fumes by means of a glass water-jet vacuum pump connected by rubber tubing to an S-shaped adapter tube, one end of which is inserted like

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a hook in the neck of the digestion flask (*2*). As soon as the reaction subsides add 10 ml. of perchloric acid and heat moderately until the solution chars or darkens and begins to clear again; then heat strongly until dense white fumes of sulphur trioxide appear near the bottom of the flask. If the solution does not clear after charring, add 0.5 ml. more of perchloric acid and heat until the white fumes of sulphur trioxide appear. A glass bead may be used to prevent bumping. Repeat the addition of 0.5-ml. portions of perchloric acid and the heating, if necessary, until a clear water-white or pale-yellow solution is obtained; then continue heating until the solution is evaporated to about 5 ml.

Cool and dilute the solution to about 15 ml. and carefully neutralize to a pH slightly over 7.0 with 1:1 ammonia, using a small piece of litmus paper for an indicator. If a precipitate separates at this point, filter the solution through acid-washed paper and rinse the flask and filter paper several times with a few milliliters of distilled water. Add 1 drop of phosphoric acid and 5 ml. of tetraethylenepentamine to the combined filtrate and washings; then dilute to 50 ml. and mix thoroughly.

Measure the transmittancy of the solution immediately, using a light filter showing maximum transmission at 595 to 640 mmu and a solution cell 50 mm. in length. (The solution can be diluted to 100 ml., if necessary, in which case a 100-mm cell should be used for the transmittancy measurement to avoid lowering the sensitivity.)

The concentration of copper in the sample can be read directly from a calibration curve formed by plotting the copper content of the standard solutions, in terms of parts per million based on the weight of test sample, against the transmittancies of the standard solutions. For example, standard copper solutions containing 1.0, 2.0, and 3.0 ml. of the diluted copper solution (0.1 gram per liter) and prepared according to the prescribed procedure will correspond, respectively, to 10, 20, and 30 p.p.m. of copper on the basis of a 10-gram sample. These values, when plotted as ordinates and the transmittancies as abscissas on semilogarithmic paper, will form a straight line from which the copper content of test samples can be read directly. No blank correction is necessary if the standard solutions are treated the same as test samples.

RESULTS AND DISCUSSION

Cellulosic material can be ashed by either wet or dry-ashing methods, but the wet-ashing method is a more convenient and positive way to bring all of the copper into solution. The digestion of a 10-gram sample can be completed in about 30 min. to 1 hr. and seldom requires more than 10 ml. of perchloric acid.

Table I. Transmittancy of Standard Copper Solutions

Volume of dilute copper solution used (0.1 gram per liter), ml.	*Weight of copper, mg.*	*Equivalent concentration for 10 grams of sample, p.p.m.*	*Transmittancy (cell 50 mm., light wavelength 620 mmu), %*[a]
0	0	0	99.3
1	0.1	10	94.5
2	0.2	20	88.5
4	0.4	40	78.3
8	0.8	80	61.5
12	1.2	120	49.5

[a] Compared with distilled water.

Table II. Recovery of Copper Added to Commercial Pulps

Pulp	*Copper added, p.p.m.*	*Total copper found, p.p.m.*	*Copper recovered, p.p.m.*
A	0	6	..
A	0	6	..
A	0	6	..
A	3	8	2
A	3	8	2
A	6	10	4
A	6	12	6
A	6	11	5
A	6	11	5
A	15	21	15
A	15	21	15
B	0	10	..
B	0	10	..
B	50	61	51
B	100	110	100
C	0	5	..
C	50	54	49

Certain precautions are necessary in handling and storing perchloric acid (*3*) and in digesting the sample. The preliminary oxidation with nitric and sulphuric acid is essential to avoid danger of fire or explosion when the perchloric acid is added. However, the perchloric acid can be added without danger as soon as the vigorous evolution of nitrous oxide subsides. Prolonged heating without perchloric acid makes the digestion difficult and may increase the amount of the acid required to complete the oxidation. It is advisable to conduct the digestion under a hood since some of the fumes may not be collected by the water-jet pump. Use of the pump, however, will prevent condensation of the acid fumes in the hood and flues.

The copper-tetraethylenepentamine complex is stable for at least 1 week at pH above 7.0, but it is less stable after the phosphoric acid is added; hence it is necessary to measure the transmittancy of the solutions as soon as possible after addition of the tetraethylenepentamine reagent.

Although some ions interfere with the color reaction of copper with tetraethylenepentamine (*1*, *4*), it appears they either are not present in wood pulps in appreciable quantities or are removed by the procedure described.

The transmittancy of standard copper solutions prepared by the procedure described above and the equivalent concentrations for a 10-gram sample are given in Table I. Obviously, a larger sample could be used to obtain higher sensitivity and accuracy, but the amount of acids required and the digestion time would be increased in proportion to the size of the sample.

The copper content of three commercial pulps and recovery of added copper given in Table II indicate that the accuracy of the method is about 1 p.p.m. for the range of 5 to 100 p.p.m. of copper in the pulp. An accuracy of this order is believed satisfactory for papermaking or for dissolving pulps.

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ASSOCIATION NEWS AND EVENTS

Corrugated Containers Conference

Assisted by the TAPPI Chicago Section the Corrugated Containers Committee will sponsor a two-day conference on corrugating problems at the Schroeder Hotel, Milwaukee, Wis., on Nov. 20–21, 1952.

The first day will be devoted to papers and panel discussions and will highlight the results of the combiner questionnaire, the most ambitious industry survey yet made on corrugated combining.

The morning of the second day will be spent in a tour through the corrugating departments of the Cornell Paperboard Products Co., The Downing Box Co., and the Gaylord Container Corp.

Following is the final program:

THURSDAY, NOVEMBER 20

8:00 a.m. *Registration*, Fifth Floor Foyer

9:00 a.m. *General Session* (East Room) W. R. Tobin, American Boxboard Co., Grand Rapids, Mich., Moderator

1. "Résumé of Combiner Questionnaire," by H. T. Scordas, Union Bag & Paper Corp., Savannah, Ga.
2. "Problem Analysis," by V. V. Vallandigham, Potlatch Forest Industries, Lewiston, Idaho.
3. "Warping and Washboarding," by C. R. Vander Meulin, Penick & Ford, Ltd., Cedar Rapids, Iowa.

2:00 p.m. *Meeting resumed*

4. "High-Low Corrugations," by F. E. Skiver, American Boxboard Co., Grand Rapids, Mich.
5. "Corrugator Delivery Tables," by A. F. Sperling, Downing Box Co., Milwaukee, Wis.
6. "An Operator Designs a Corrugator," by Keith W. Max, Robert Gair Co., Uncasville, Conn.

5:30 p.m. *Social Hour* (Crystal Ballroom)

6:30 p.m. *Dinner* (Crystal Ballroom)
Speaker to be announced

FRIDAY, NOVEMBER 21

8:30 a.m. *Plant Tours*
Cornell Paperboard Products Co.
Downing Container Corp.
Gaylord Container Corp.

Annual Meeting

The Annual Meeting of the Technical Association will be held at the Commodore Hotel, New York, N. Y., on Feb. 16–19, 1953.

Room reservations should be made direct with the Commodore or other New York City hotels.

A tentative program will be published in the December issue of *Tappi*.

Local Section Meetings

Pacific: Nov. 18, 1952, at Camas, Wash. This will be the annual engineering meeting. The research laboratory of the Crown Zellerbach Corp. will be open for inspection. The Jan. 13, 1953, meeting will be held at the University of Washington, Seattle, Wash. The technical program will be devoted to the consideration of routine mill control methods.

Lake States: The Jan. 13, 1953, meeting of the Lake States Section will be held at the Conway Hotel. "Stainless Steel in the Paper Industry" will be discussed.

Delaware Valley: The next meeting of the Delaware Valley Section will be held at the Engineers Club, Philadelphia, Pa., on Dec. 4, 1952. Hugo H. Hanson, President of W. C. Hamilton & Sons, Miquon, Pa., will discuss "Problems of Managing a Paper Mill."

On Jan. 22, 1953, there will be a panel discussion of "Development of Paper Properties." Ward D. Harrison, Vice-President of the Riegel Paper Corp., Milford, N. J., will be the moderator.

Kalamazoo Valley: The Kalamazoo Valley Section will meet at the Harris Hotel, Kalamazoo, Mich., on December 4. The subject of the meeting will be "Production Control in Printing and Quality Control in Pulp Manufacturing."

The annual joint meeting of the Section and the Michigan Division of the Superintendents Association will be held at the Hotel Harris on Jan. 15, 1953.

New England: The next meeting of the New England Section will be held at the Roger Smith Hotel, Holyoke, Mass., on Nov. 21, 1952. This meeting has been designated "Miss Helen Kiely Night."

Ohio: The Ohio Section will meet at the Manchester Hotel, Middletown, Ohio, on Nov. 18, 1952. "Electric Distribution Systems in Paper Mills" will be the subject of the meeting.

Empire State: The Empire State Section is divided into five districts, each of which hold monthly meetings.

Northern District: The Northern District will meet at the Hotel Woodruff, Watertown, N. Y., on Dec. 11, 1952. A speaker from the Forest Products Laboratory, Madison, Wis., will discuss "The Utilization of Hardwoods."

On Jan. 8, 1953, the District will meet at the Woodruff. Wilfred Galley of E. B. Eddy Co., Hull, P. Q., will discuss "What Holds Paper Together."

Central District: The Central District will meet at the University Club, Syracuse, N. Y., on Dec. 5, 1952. Hilliard D. Cook of Sweet Bros. Paper Mfg. Co., Phoenix, N. Y., will talk on "Beating—An Old Angle."

On Jan. 9, 1953, the District will meet in Syracuse. Ernest Reed of Syracuse University will discuss "Control of Microbiological Growths in Pulp and Paper Mills."

Western District: The Dec. 3, 1952, meeting of the Western District will be held in the Cafeteria of Moore Business Forms, Inc., Niagara Falls, N. Y. The meeting will feature a visit to the printing plant of the company.

Lake Erie: The Lake Erie Section will meet at the Hotel Carter, Cleveland, Ohio. This will be the packaging meeting. J. M. Cowan, Managing Director of the National Flexible Packaging Association, will talk on "The Battle of the Films." Mr. Cowan was formerly with the Dobeckmun Co. and the Cellophane Division of du Pont. His talk will review the history, availability, and unique properties of each of the types of commercially available transparent films used for packaging purposes. (November 21, 1952).

Southeastern: The Jan. 23, 1953, meeting of the Southeastern Section will be held at Charleston, S. C.

Training and Education Survey

Many pulp and paper mills in the United States will soon

receive from the TAPPI office an inquiry requesting the listing of training programs for employees that are now being conducted, that have been conducted in the past few years, or that are expected to be conducted in the near future. The inquiry will be self-explanatory and can be answered in a very few minutes.

This survey is the first step taken by the reactivated Training and Education Committee, which is to present to the industry recommendations on the training of supervisory employees, including executives; of prospective supervisory employees—leadmen and others from whom future supervisors may be expected to be drawn; and of nonsupervisory employees in production, maintenance, technical, and clerical occupations. In its work the committee will study such matters as the means of determining training needs, the development of training content, the effective methods of conducting training, and the means of measuring the results of training.

It is expected that the findings of the committee will contribute materially to the development of the human resources of the industry. The cooperation of the various plants will greatly assist the committee in its work.

At its first meeting in Savannah, Ga., on September 9 and 10, the committee laid the plans for carrying out its assignment and formed itself into two subcommittees, one to study supervisory and presupervisory training and the other to study nonsupervisory training. The committee is composed of: for supervisory and presupervisory training, D. D. McKinney, Educational Supervisor, Hammermill Paper Co., co-chairman, George Arthur, Training Coordinator, Champion Paper and Fibre Co., and L. M. Lenzi, Training Director, The Mead Corp.; for nonsupervisory training, J. A. Luke, Personnel Manager of the Charleston, S. C. plant of West Virginia Pulp and Paper Co., co-chairman, G. H. Gallaway, Asst. Resident Manager of the Camas, Wash., plant of Crown Zellerbach Corp., and J. H. Groves, Training Director, Union Bag and Paper Corp. J. H. Groves is chairman of the full committee.

Deinking Monograph

During the past year there has been widespread attention paid to the use of wastepaper to replace and supplement wood pulps. As a result, the tonnage of wastepaper consumed in 1951 was the largest in history.

Recognizing this interest the TAPPI Deinking Committee has felt that it is now time to begin the preparation of a Monograph on the Deinking of Wastepaper. At a committee meeting at Niagara Falls, Ont., in June a tentative outline was drawn up. Since that time a number of individuals have been asked and have agreed to contribute to the Monograph.

These include S. D. Wells, Institute of Paper Chemistry; Haskell Stovroff, Buffalo Waste Paper Co.; D. M. Dickson, Penn Paper and Stock Co.; Merle Wilkins, Kalamazoo Paper Co.; John Clouse, Oxford Miami Paper Co.; H. E. Nelson, French Paper Co.; Rolfe C. Bradbury, the Mead Corp.; Harold Morrison, Consulting Engineer; F. C. Goodwill, St. Regis Paper Co.; Paul Hansen, Bergstrom Paper Co.; A. J. Palladino, National Council for Stream Improvement; and A. M. Altieri, Tileston and Hollingsworth Co. Others will be announced at a late date.

There are several sections for which the committee would be pleased to secure additional contributors. Any individual desiring to contribute in the work would be welcome and should write promptly to the chairman, J. J. Forsythe, International Paper Co., Niagara Falls, N. Y. A committee meeting will be held in New York during the February TAPPI Convention.

The following is a tentative outline of the deinking monograph:

I. Historical Review of Deinking and Statistics on Deinking.
II. Properties of Reclaimed Wastepaper.
III. Gathering and Sorting of Wastepaper by Packers.
IV. Grade Classifications of Wastepaper.
V. Mill Testing of Wastepaper.
VI. Sorting Practices in Wastepaper Use.
VII. Theory of Deinking—Effect of Chemical Agents Used Upon Nonfibrous Ingredients.
VIII. Defibering and Ink Removal.
IX. Screening and Washing of Deinked Stock. Removal of Foreign Materials.
X. Alkali and Heat Recovery from Deinking Operations.
XI. Bleaching of Deinked Stock.
XII. Handling of Deinked Waste Effluents.

Seventh Engineering Conference

The Seventh Engineering Conference of the Technical Association of the Pulp and Paper Industry was held at the Morrison Hotel, Chicago, Ill., Sept. 13–16, 1952. The General Chairman, C. J. Sibler of the West Virginia Pulp & Paper Co., opened the meeting with a brief outline of the objectives of the Engineering Division after which the chairmanship was turned over to H. F. Parker, New York & Pennsylvania Co., Chairman of the Mill Maintenance and Materials Committee.

The first speaker to be introduced was E. L. Lockman, United States Rubber Co. who spoke on the "Care and Maintenance of Rubber Covered Rolls in the Pulp and Paper Industry." The fact that the subject of the care and maintenance of rubber covered rolls does not have the glamor or the attention-attracting appeal that discussions of new products or new processes possess, does not in any way diminish its importance of the problem of obtaining the maximum operating performance from items which in themselves, while expensive, are also vital to the continued and efficient operation of machines representing large investments.

The rules which were presented, while simple and based on common knowledge should be constantly brought to the attention of all concerned. They may also be incorporated in any training program for new employees or refresher talks or discussions with old employees if such meetings are part of a mill's maintenance or training program.

This paper was followed by "The Engineer's Approach to Accident Prevention" by George W. Harper of the University of Illinois.

While there has been much progress in accident prevention in industry in this country, there is still much to be done. The accident rate in the pulp and paper industry has decreased since 1944 but is still above the average for all industry. Smaller mills have a higher rate than larger mills where organized safety programs are in effect.

If additional gains are to be made it was the speaker's conviction that they will come through the engineering approach. For many years management and employes have heard that 80% of all injuries were caused by human failure and 20% were caused by physical hazards. This fallacy was attacked, and cases were given showing that the hazard factor is often overlooked. It was pointed out that usually both factors are present and that the basic attack must be on hazard elimination through engineering applications.

The engineering approach to accident prevention presents a challenge to every member of TAPPI—those in large plants

A. P. Schnyder, Ebasco Services, Inc.; G. W. Harper, University of Illinois; H. F. Parker, New York & Pennsylvania Co.; J. A. Holekamp, American Pulpwood Assoc.; E. L. Lockman, United States Rubber Co.; and C. J. Sibler, West Virginia Pulp & Paper Co.

90% OF ALL THE PAPER SCREENED IN NORTH AMERICA PASSES THROUGH

BIRD SCREENS

Bird Screens are the last barrier between dirt and the stock that flows onto the paper machine wire.

Bird Screens comb out the fibres and deliver a homogeneous mixture of clean fibres and water, free of lumps, strings and slime.

Bird Screens have the capacity to deliver all the stock the paper machine can take and at the same time provide gentle, efficient screening action.

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to increase their accident prevention work and cooperate with their safety departments. Those members in smaller mills have a greater opportunity in that they can initiate a sound safety program based on engineering principles.

After these two papers the program was conducted by A. P. Schnyder of Ebasco Services, Inc., Chairman of the Materials Handling Committee. There was only one speaker, James A. Holekamp of the American Pulpwood Association. This paper "The Preparation, Handling, and Transportation of Pulpwood East of the Rockies" covered a generalized picture of the material handling techniques currently in use by the pulpwood industry east of the Rocky Mountains. The cutting, skidding, truck loading, truck unloading, and long distance hauling phases of the production of pulpwood were each presented with photographs and a discourse of the equipment and methods employed by the various pulpwood producers in the South, Lake States, and Northeast regions.

During the noon hour Monday and again on Tuesday the committees held luncheons at which they discussed their individual programs for the future.

At 2 p.m. the Drying and Ventilating Committee Chairman, M. L. Barker, Beckett Paper Co., resumed the formal program by introducing A. J. Cirrito of the Lukens Steel Co. who gave his paper, "An Evaluation of Drying Concepts."

Numerous theories and some experimental data have been published on the factors which influence the drying of paper. Yet, progress in drying is still considered to lag behind advancement at the wet end. Higher speeds and the desire for improved sheet quality require more thorough considerations than the mere increase of steam pressure or the addition of driers.

The major factors in paper drying were discussed to show how they relate to the fundamental problem. Constructive criticism was offered for some widely accepted hypothesis. Analogous heat and mass transfer data in fields other than paper drying were examined together with new concepts for more efficient use of the mechanism of drying.

While some of the factors may be evaluated in the laboratory or pilot plant machine, many are peculiar to individual papers and speeds. These must be solved in the mill with the understanding cooperation of the papermaker. Not only should a broad analytical viewpoint be encouraged, but advanced techniques for field investigations must be developed.

In "Follow the Rules for Proper Steam Blast Coil Installation and Avoid Costly Trouble," the next paper, Richard G. Lubinsky of the Trane Co., stated that most troubles encountered with steam coils on industrial applications can be traced back to improper installation. In just about every instance, the installation violates long established rules—rules that have been formulated only after unsatisfactory field experience.

There are two all important general rules that must be followed if satisfactory operation is to be expected: (1) air must be thoroughly vented from heat transfer equipment and (2) condensate must drain freely into return lines.

Any system on which these two things are not accomplished will be troublesome because the capacity of the transfer equipment will be greatly reduced and early failures may be expected because of: (1) corrosion, (2) rupture due to a difference in expansion of tubes, (3) breakage due to excessive pressures from water hammer, and (4) rupture due to freezing.

Common errors in installation which result in the previously mentioned difficulties are: (1) improper pitch of the coil and piping, (2) improper trapping or the use of traps that are not sufficiently large to handle requirements, (3) use of one trap on a number of coils arranged for series air flow, (4) selection of the wrong type of heating coil, (5) no provision for expansion of piping and coil tubes, and (6) use of the wrong combination of controls, traps, and piping system.

Many operating engineers do not realize the seriousness of these errors only because they have not given thorough consideration to fundamentals. A detailed study of what happens in the system when each of the errors is encountered will readily explain why they must be avoided.

It may cost slightly more to make a carefully engineered installation following all the rules of good installation practice but it will prove economical in the long run. The cost of making repairs and of lost production during shutdowns can be tremendous.

The three committees, sponsoring the first sessions then held open meetings at which the various papers and other matters of interest to the committee were discussed.

The Annual Engineering Division Luncheon at the Seventh Engineering Conference, Chicago, Ill., Oct. 15, 1952

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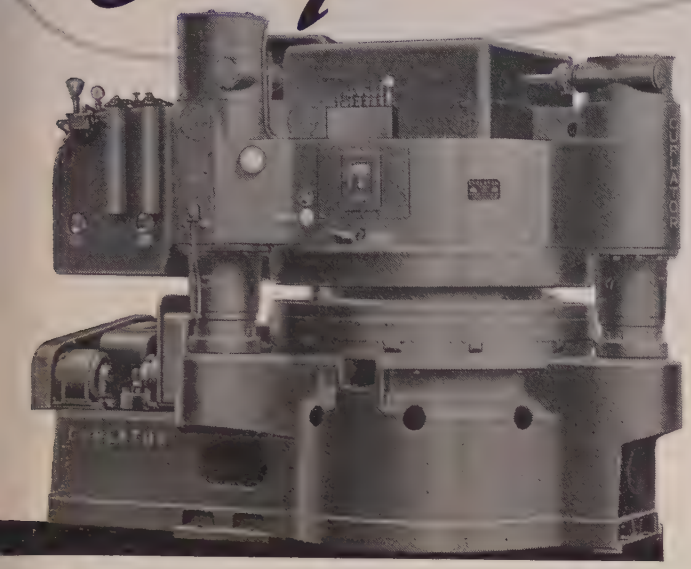

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ELIMINATE ALL FIBER BUNDLES AND REDUCE DIRT

IMPROVE PULP QUALITY

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AND STILL RETAIN FREENESS AND FIBER LENGTH

Curlators are increasing pulp yield as much as 10% in commercial operation, where sulphite pulp is used in unbleached papers. When sulphite pulp is used in bleached papers the increase in yield is not quite as great.

Because Curlation does not reduce fiber length the rejections from fine screens can be increased to obtain improved cleanliness, and these rejects can then be Curlated and returned to the system for the production of additional No. 1 pulp.

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†T. M. Reg.—Curlator Corporation, Rochester, N. Y.

M. Jacobs, Chas. T. Main, Inc.; W. F. Hathaway, Kalamazoo Vegetable Parchment Co.; C. A. Young, Riegel Paper Corp.; W. C. Bloomquist, General Electric Co.; J. R. Lientz, Union Bag & Paper Corp.; R. W. Foster, Champion Paper & Fibre Co.; H. F. Parker, New York & Pennsylvania Co., Inc.; M. L. Barker, Beckett Paper Co.; K. P. Geohegan, Howard Paper Mills, Inc.; and A. J. Miller, Jr., The Mead Corp.

W. A. McKenzie of the Simpson Logging Co. led a group on the harvesting and handling of smaller sizes of pulpwood from the forest to the wood yard in the Materials Handling meeting and R. Q. Conner, Crossett Paper Mills, on the tool room versus the store room for maintenance tools in the Mill Maintenance and Materials meeting.

The Steam and Power Session was first on the program Tuesday, October 14, and H. R. Arnold, Riegel Carolina Corp., the Chairman, introduced J. D. Yoder of the Permutit Co. who presented his paper "Hot Lime Zeolite Treatment and Demineralization of Boiler Feed Make-up."

Paper mills, as well as other industrial plants requiring process steam and considerable power, can manufacture their power as a by-product of process steam at a very low cost. This usually requires high pressure boilers which demand first class boiler feed water treatment. During recent years methods of boiler treatment have greatly improved and are available for the most exacting needs.

This paper discussed two modern types of boiler feed water treatment that are well suited to the paper industry. One of these is a two stage hot lime-zeolite treatment and the other is demineralization.

Each of these types of boiler treatment was briefly described and the particular advantage of each type of treatment as well as comparative study of treatment costs and costs of chemicals was given.

The next speaker was Frank X. Gilg of the Babcock & Wilcox Co. who spoke on "Packaged Steam Generators in the Paper Industry."

A "packaged steam generator" is a standardized, shop-assembled boiler, equipped and shipped complete with fuel-burning equipment, mechanical draft equipment, automatic controls with fuel and low-water cut-outs, and other accessories.

There are "fire-tube" types and "water-tube" types. This paper described a water-tube type and showed that it really is an extension of the integral-furnace series, in which boiler and water-cooled furnace are designed as an integral unit, with over-all dimensions of length, width, and height limited to meet common-carrier (rail, truck, water) specifications on weight and clearances.

For sizes to generate up to 28,000 lb. of steam per hour and moderate pressures to 250 p.s.i., packaged steam generators are being used more and more in the paper industry because low fuel costs, low maintenance and operating costs, compactness, portability, simplicity, reliability, and safety in operation are necessities in this progressive industry.

Typical examples with pictures of modern installations in paper mills were described with their salient features, operating data, and fuel costs. Automatic combustion control systems were described to show the emphasis given to safety and simplicity in operation.

The Mill Design and Economic Aspects Session followed the Steam and Power Session "Government and the Pulp and Paper Industry in the United States" was the subject of the talk by Allan Hyer, Bagley & Sewall Co., who was formerly with the National Production Authority. Mr. Hyer indicated the reasoning which led to the creation of the National Production Authority for the control of materials and for the planned expansion of industry during the time of defense build-up such as now exists. He showed the necessity of combining the operations of industry and Government to accomplish those objectives, particularly as they relate to the pulp and paper industry, and to meet any requirement that might occur in the future during an emergency period. The manner in which the original program was designed and its application to the pulp, paper and board sector, the incentives offered by Government to promote and encourage expansion on the part of private industry to meet this program, and the important part played by individuals from private industry in the organization and implementation of this program was mentioned. Details concerning the actual operation of the Pulp and Paper Divison of NPA were given. As a final word, the present status of the program was given with an indication of what industry can expect as a result of this Government control in the future.

The afternoon was devoted to a session sponsored by the Chemical Engineering Committee followed by discussion periods conducted by that committee as well as the Mill Design and Economic Aspects and the Steam and Power Committees.

J. R. Lientz, Chairman of the Chemical Engineering Committee, introduced Nicholas Shoumatoff, West Virginia Pulp & Paper Co. The paper "Digester Corrosion Measurement," presented the results of a study of corrosion in no. 4 digester at the Luke, Md., mill of the West Virginia Pulp &

C. J. Sibler, West Virginia Pulp & Paper Co.; G. W. E. Nicholson, Union Bag & Paper Co.; W. J. Hemphill, Johns Manville Corp.; G. H. Pringle, The Mead Corp.; P. A. Forni, Mutual Security Agency; H. J. Perry, Paper Trade Journal; K. F. Mackenzie, Eastman Kodak Co.; J. D. Lyall, Armstrong Cork Co.; R. G. Macdonald, TAPPI; W. Wyburn, Federal Paperboard Co.; and R. P. Price, Hammermill Paper Co.

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C. W. Parsons, Republic Flow Meter Co.; R. E. Bergstrom, Swenson Evaporator Co.; W. C. Bloomquist, General Electric Co.; J. A. Minser, Crane Co.; G. H. Pringle, The Mead Corp.; G. W. E. Nicholson, Union Bag & Paper Corp.; and W. C. Reynolds, Journal-Times of Racine, Wis.

Paper Co. based on three sets of semiannual readings with the Audigage thickness tester. The emphasis of the present paper was on the procedure of analyzing the inspection results, rather than attempting to generalize from the specific findings. To determine and evaluate the magnitude and variations of corrosion indicated by the readings, the records were analyzed statistically by the analysis of variance and linear regression procedures.

It was found that local variations in plate thickness are significant but that variation of corrosion rates is significant only in the vertical direction. Maximum corrosion rates were found near the knuckle section of the tophead with an estimated remaining service life of 3 yr. at this location, but with lower corrosion rates and a greater life expectancy both above and below the critical area. It was also found that the general corrosion rate increased significantly during the second inspection period. It was felt that this study establishes the validity of the Audigage inspection procedure as a powerful tool for the study of digester corrosion.

H. O. Teeple of the International Nickel Co. then gave a summary of the report "Corrosion of Alkaline Pulping Digesters—Analysis of Corrosion Questionnaires" by R. S. Peoples and G. L. Ericson, Battelle Memorial Institute, which appeared in the September, 1952, issue of *Tappi*.

This report contains the results of a study of information obtained from replies to a questionnaire sent to 63 pulp mills in regard to corrosion problems in the industry. The information revealed that corrosion has been significantly greater in digesters installed during the last 10 yr. For example, of the digesters in operation during this period, 24% showed corrosion rates of over 100 mils per yr., while essentially 100% of those in operation prior to this time showed rates of less than 50 mils per yr. The data submitted, though thoroughly screened, were not adequate to establish whether the observed trend was the result of (1) changes in operational and processing procedures, (2) type and quality of steel, or (3) as yet unidentified variables. To determine the reasons for this trend, it would be necessary to: (1) obtain more precise and detailed information from either all the mills or selected mills in the industry with respect to processing procedures and data, (2) obtain more comprehensive and accurate corrosion information in regard to rates, type, and location of attack, (3) obtain a more complete chemical analysis of metal samples from digesters undergoing corrosion examination, (4) make a complete microscopic examination of representative metal samples, both from digesters giving long life and from those showing high rates of attack, (5) secure more comprehensive data with respect to metal processing and fabrication on individual digesters.

In the Steam and Power Discussion D. F. Langenwalter, General Electric Co., opened the meeting with a paper "Relaying Protection for Operating an Industrial Plant in Parallel with the Utility System" which was intended to serve as an introduction to the topic of relaying the tie between the industrial plant and the utility. This topic is becoming more and more important as utilities and paper mills establish electrical ties for greater continuity of service.

The electrical operating problems associated with such tie operation were described and proper relaying was suggested for various operating conditions. Protection is necessary to avoid loss of production and to prevent damage to motors and generators.

The reclosing speeds of breakers and various types of relays for sensing electrical disturbances were disclosed.

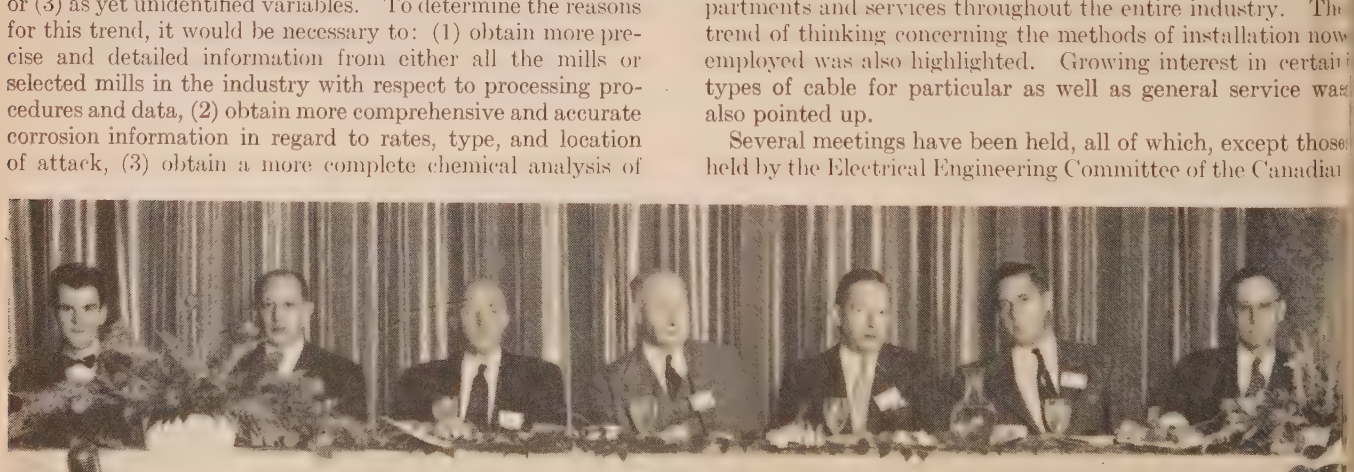
On Tuesday evening there was a social hour which preceded a dinner given by the allied industries.

R. W. Foster, Champion Paper & Fibre Co., Chairman of the Electrical Engineering Committee, opened the Wednesday sessions.

A. O. Mortenson, St. Regis Paper Co., submitted recommended electrical cable specifications for the pulp and paper industry.

From a questionnaire submitted to the industry in May 1951, a wealth of information was received, recorded, and studied, separately and jointly, by TAPPI, the Insulated Power Cable Engineer's Association, and the Canadian Pulp and Paper Association, in conjunction with the Canadian cable manufacturers. That study revealed what was preferred, from what the market afforded, for the various departments and services throughout the entire industry. The trend of thinking concerning the methods of installation now employed was also highlighted. Growing interest in certain types of cable for particular as well as general service was also pointed up.

Several meetings have been held, all of which, except those held by the Electrical Engineering Committee of the Canadian



D. H. Putnam, General Electric Co.; C. J. Sibler, West Virginia Pulp & Paper Co.; J. W. Hemphill, Johns Manville Corp.; R. G. Macdonald, TAPPI; W. R. McNally, Link-Belt Co.; J. R. Lientz, Union Bag & Paper Corp.; and M. Jacobs, Chas. T. Main, Inc.

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For Fine Papers

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For a smaller system where fine dirt and removal of abrasive materials are the main objectives—manual care is required for this system.

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For larger systems where removal of abrasive dirt, bark specks, shives and similar materials are the main objectives. This system is *entirely automatic* and requires *no manual dumping*.

Patented bleed principles particularly effective for removal of light foreign material.

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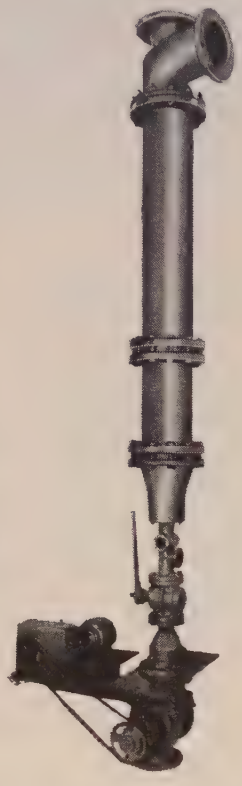

8" AND 10" VORTRAPS WITH AUTOMATIC REJECTS VALVE

For Rough Papers

FOR WASTE PAPERS

The 6", 8" and 10" Vortraps are very effective in removing abrasive dirt, glass, rubber, sand and similar foreign materials.

The new 6" Vortrap is specifically designed for use on board mill liner stocks.

Operation can be standard by use of waste receiver which requires manual dumping or *completely automatic* through use of our newly developed automatic discharge valve.

These units are of particular value to Board and Pulp Mills and DE-INKING SYSTEMS.

4" VORTRAP

GENERAL:

1. *Screen Tailings* . . . Effectively cleaned by Vortraps.
2. *Pulp Mill Installations* . . . Large volumes of stock may be cleaned effectively by a combination system of the larger and smaller Vortraps.
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4. The elimination of solids and foreign matter from *slurries* and *water* is another of the many uses of the Vortrap in its different sizes.

1½" VORTRAP

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Monohearth bark burning furnace for the disposal of *wet bark*. Ore roaster for the production of SO_2 gas and other furnace applications.

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The Mill Design and Economic Aspects Committee

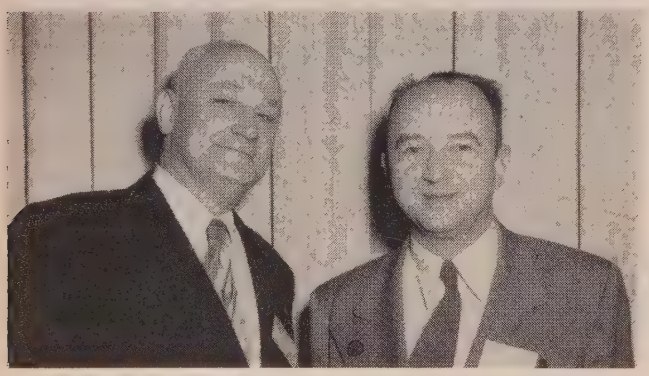
Pulp and Paper Association, have been reported in *Tappi.*

Specifications have been written to cover the entire industry—branch by branch, department by department, and service for service. They were presented for approval.

An attempt has been made to cut the number of cable types and sizes to a practicable minimum without surrendering any of the best procurable materials from those available and yet meet the needs as stipulated by the recorded data from the questionnaire of May, 1951.

Next on the program was the final report on press section load requirements by R. R. Baker, Westinghouse Electric Corp.

TAPPI Data Sheets 145 G, H, I have been prepared giving recommended power constants for the various sections of the paper machines. To continue the effort of establishing the proper value of power constants for the various sections



A. O. Mortenson, St. Regis Paper Co. and R. W. Foster, Champion Paper & Fibre Co.

of the paper machine, the Electrical Engineering Committee decided, in this particular instance, to make a more detailed study of the press section. In making the detailed study, questionnaires were submitted to a number of mills operating sectional electric drives where accurate information was obtained on the power requirements and operating conditions of various press sections. From a study of this power data, recommendations have been made for modification and corrections of the power constants appearing on the TAPPI Data Sheets. The full report has been published in the September, 1952, issue of *Tappi.*

The last session of the morning was devoted to the work of the Hydraulics Committee and K. W. Mackenzie, Eastman Kodak Co., presented Richard E. Durst, who with Lyle C. Jenness, prepared the summary, "Progress Report on the Relationship of a Stock Shear Tester to Head Losses in the Flow of Pulp Stock in Pipes," which was a review of

The Electrical Engineering Committee

The Chemical Engineering Committee

the present status of the hydraulics project at the University of Maine sponsored by the Hydraulics Committee of TAPPI.

A pulp stock flow system has been installed and some exploratory tests have been made. The system consists of two 2200-gal. stock chests, a high consistency stock feeder and pump, appropriate valves, and spiral welded wrought iron pipe to provide a 60-ft. straight section of $6^5/_8$ in. o.d. 0.141-in. wall, pipe for the pressure drop measuring section. The test section has pressure taps located so that measurements may be made over 10, 20, 30, or 40-ft. lengths of pipe with calming sections of 10 ft. minimum length before and after pressure taps. Pulp stock for the system is prepared in a 3-ft. Hydropulper. Pressures at the test points are read from calibrated test gages protected from the stock by oil-filled diaphragm pressure seals.

A shear tester has been designed and constructed. The instrument may be described as a large scale viscosimeter with a rotating member which delivers torque through the stock being investigated to the wall of the stock containing

The Materials Handling Committee

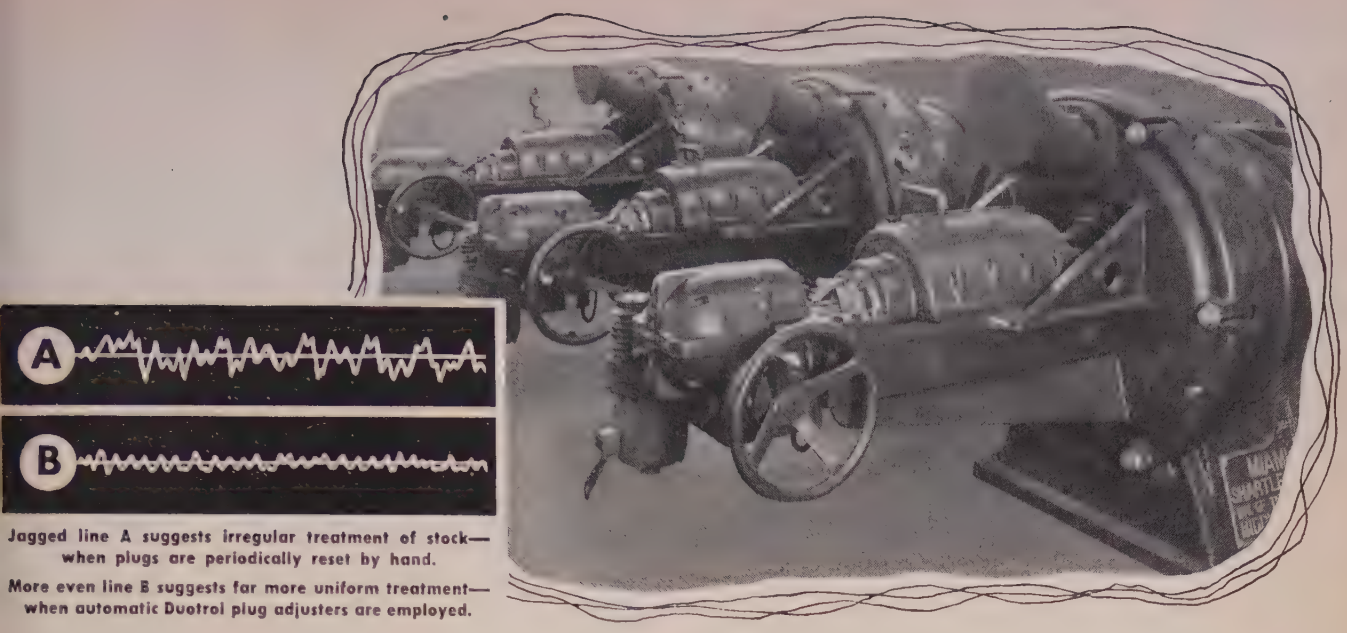
cup which is suspended on ball bearings and is free to rotate. The cup rotation is restrained by balancing weights, whose mass is a measure of the torque delivered to the cup by the stock.

The shear stress, expressed in pounds per square inch, has been measured on four stocks over a range of consistencies from about 0.5 to 5.0% moisture-free stock. The shear stress, when plotted versus rotor peripheral speed in feet per second indicates that for any given stock of consistencies below 1.0% the shear stress is nearly independent of rotor speed. The data are incomplete for higher consistencies but indications are that the shear stress is a function of consistency and velocity for any given stock.

The investigation is being continued and the data taken in the shear tester on the stocks being used in the pulp stock flow system are being studied for a correlation of shear stress with pipe friction loss.

The Engineering Division Luncheon was held in the Mural Room of the Hotal Morrison. C. J. Sibler was toastmaster

The Hydraulics Committee



Jagged line A suggests irregular treatment of stock—when plugs are periodically reset by hand.

More even line B suggests far more uniform treatment—when automatic Duotrol plug adjusters are employed.

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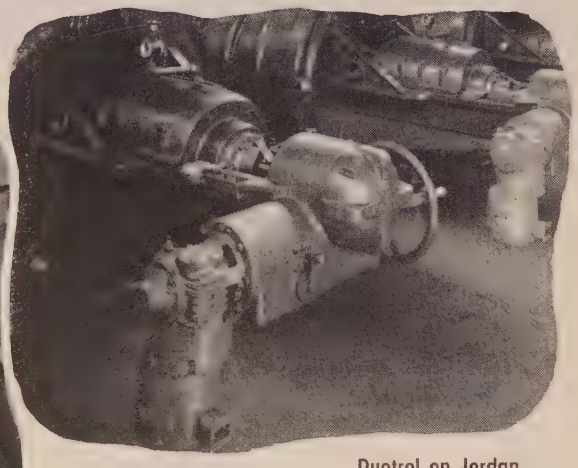

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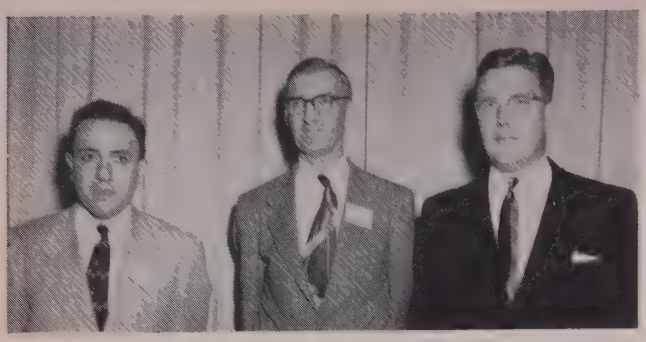


A. J. Cirrito, Lukens Steel Co.; M. L. Barker, Beckett Paper Co.; and T. W. Ernst, The Trane Co.

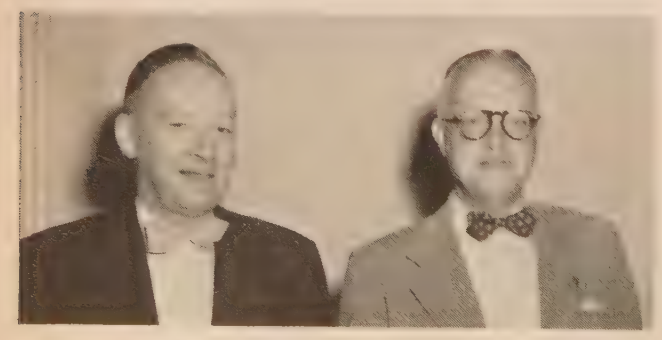
and introduced the guests at the head table. Among those introduced was G. W. E. Nicholson of the Union Bag & Paper Corp. In behalf of the Engineering Division Mr. Nicholson presented J. W. Hemphill of the Johns-Manville Co., the first general secretary of the Division, with a beautiful watch.

The principal address was: "People Have a Place in Your Planning," by A. J. Miller, Jr., Assistant Director of Industrial Relations, the Mead Corp., Chillicothe, Ohio. This appears in the forward section of this issue of *Tappi*.

At 2:30 p.m. the session resumed with the program of the Engineering Research and Machine Design Committee, J. D. Lyall, Armstrong Cork Co., Chairman. The first paper was "Flow in 'Converging Nozzle' Type Slices—Can Negative Pressure Be Present?" by J. B. Gough of The Mead Corp. (Sept. 1952 issue of *Tappi*.)

This analysis investigated the pressure of the flow against a straight converging top slice plate, in an approximate manner, for any orifice or jet velocity (particularly at 300 to 600 f.p.m.), any orifice or slice opening (particularly at 0.36, 0.375, and 0.72 in.), and any convergent angle (particularly at 50, 40, 30, 20, and 10°) of the inclined top slice plate in relation to the horizontal bottom apron plate.

The results indicate that a negative pressure (i.e., below atmospheric pressure) will be present at this inclined top slice surface for a jet or approximate wire speed of 300 f.p.m. *if* in contact with the liquid (or pond) for any greater distance than 6 in. approx. (from the lip edge), when the angle of convergence is 50° (or more), or for any greater distance than 9 in. approx., when this angle is 30° (or more); thus a negative pressure will occur in a completely sealed nozzle (one having no pond or liquid-air surface), or in a sealed pond under a vacuum, if these critical lengths are exceeded. For speeds of 600 f.p.m. or more, no such negative pressure will be present, even at distances of 24 in. from the lip, unless the angle of convergence exceeds 50°, or unless a sealed pond is under a sufficient vacuum. However, with any liquid-air pond surface, it is possible that a negative pressure between the liquid and the surface of the inclined top slice plate will occur at and just below the point of intersection of pond and inclined plate surface, if the approach velocity of the pond surface is zero or very low.



W. R. McNally and H. F. R. Weber, Link-Belt Co.

H. R. Arnold, Riegel Carolina Corp.; J. D. Yoder, The Permutit Co.; F. X. Gilg, The Babcock and Wilcox Co.; and J. Gottlieb, Westinghouse Electric Co.

Two alternative methods of analysis are used, the first neglecting the effect of friction and the second taking it into account, approximately.

The added effect of the curved flow which may be present, due to the usual hinge or curve in the top plate, which will lower these pressures still further, was also briefly discussed; also the possibility of air entrainment at any liquid-air surface, such as an open or closed pond, if this surface intersects the inclined surface of the top slice plate, and the surface velocity of approach is zero or very low, or if the pond surface intersects a curve or hinge in the top slice plate. The possibly bad effect of these negative pressures (and air entrainment) on the smoothness and uniformity of the jet from the slice orifice and on the resulting formation and sheet quality is also discussed.

This was followed by "Photographic Measurement of Drainage Rate-A Method for Determining Sheet Making Rates in Cylinder Machines" by Charles F. Payne and William E. Guthrie, Eastman Kodak Co.

Definition of the exact rate of formation of a sheet on a cylinder mold has been desired by papermakers for many years. This information can now be obtained accurately by a photographic method of measuring the drainage rate through a facsimile cylinder mold section. An apparatus was described which can be calibrated to duplicate the drainage rate that exists on the particular machine under study. The cylinder machine's changing papermaking head conditions are automatically reproduced over a wire section identical to a similar area on the machine. Stock taken from the machine headbox was used in all of the tests. The white water drained through the facsimile mold section is collected and measured in a calibrated U tube which is enclosed in an air-tight windowed chamber. The air pressure within the chamber can be adjusted to a level equal to the papermaking head above the drainage area and controlled discharge of this compressed air through a calibrated orifice permits duplication of the increasing head prevailing on the cylinder machine. The drainage rate is determined by taking simultaneous motion pictures of the U tube and a high speed clock. The motion

J. R. Lientz, Union Bag & Paper Co.; N. Shoumatoff, West Virginia Pulp & Paper Co.; and H. O. Teeple, International Nickel Co.

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pictures are examined, one frame at a time, and the total volume of white water in the U tube is plotted versus time on the clock to obtain a curve of the drainage rates. Once this drainage curve has been established the amount of sheet built up can be determined for any point in the cylinder's travel.

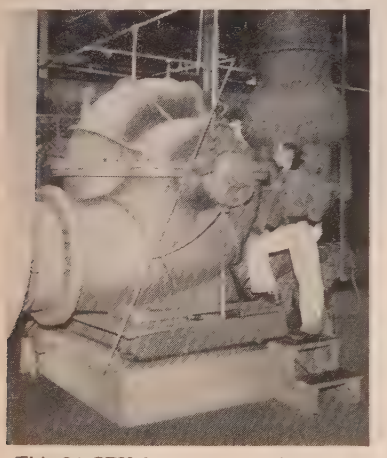
The apparatus may also be used to test the effect of various stock slowness values, consistency values, and wire meshes on the rate of sheet formaton. Other applications for this drainage measuring method will be found in the determination of the decreasing volume of stock in the annular space between the cylinder vat circle for overflow vats. Knowing this decreasing volume for various points along the vat circle the average stock velocity can be calculated for these same points. If desired the data may be used for design of vat circles producing definite stock velocity relationship with respect to the moving cylinder.

The final paper was presented by Jesse A. Jackson of the St. Regis Kraft Corp., "An Analysis of a Cylinder Vat." In making a fresh approach to so complex a device as the cylinder vat it becomes necessary to reduce each part of the problem to its simplest essentials. The prototype is an existing counterflow vat, one of five serving an old veteran of a machine which is still turning out profitable tonnages. However, they wanted to improve its ability to exceed the customers' specifications on one of the newer types of paper that have been developed in recent years. A survey of existing literature indicated that experimental test equipment would have to be rather elaborate if it were not to interfere with regular production, and there would still be considerable doubt as to the validity of the test results due to the complicated nature of the various factors involved. They were limited, therefore, to any analysis of the flow conditions step by step through the machine, based on pure hydraulic theory. They were encouraged in this effort by the knowledge that there is plenty of practical know-how at hand to prevent them from becoming lost in the nebulous clouds of abstract theory. It was fortunate also, in making the basic assumptions, to find that there was available certain published information on the percentage of formation around the wire. This variable, for which there would otherwise have to be the wildest kind of a guess, is based on some extremely simple experiments with a strap laid against the turning wire. Because of the general nature of these data, and the other simplifying assumptions that must be made, the results of the studies are chiefly of value in giving an overall picture of what is happening to the fibers at each point of their progress through the machine. Of even greater potential value, however, is the information which becomes available to the designer when he can compare, by this method the characteristics of two or more vats of similar design. The method was explained in general terms with sufficient detail of the important formulas so that calculations may be tabulated step by step in an orderly manner.

After the formal papers, the various committees having sessions Wednesday held discussion meetings. At the Electrical Engineering Committee meeting, J. L. Van Nort, of the Reliance Electric & Engineering Co. gave a summary on the "Power Requirement of Supercalenders." The others discussed the papers previously presented and their contemplated projects.

Thursday was devoted to plant visits. Many took the opportunity to visit the Chicago Stockyards, the Crane Co., and the Inland Steel Corp.

There were many who contributed to the success of this conference which was attended by about 475 engineers from all over the country, from Canada, Norway, and the Netherlands. The Engineering Division is very appreciative of their efforts and those companies which so kindly arranged plant visitations.



This 24 CFV fan pump at a Southern mill delivers 28,000 gpm of ½% stock to a paper machine.

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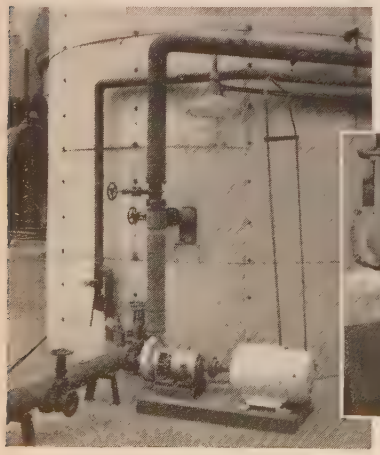
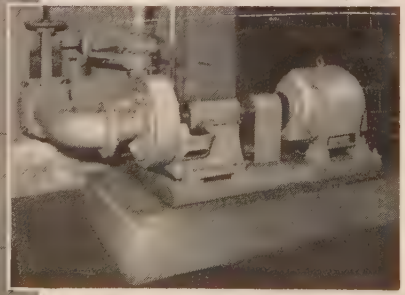


One of several I-R chemical pumps used on sizing circulation service at a paper mill.

A 4 EGV pump moving 3½% stock from a surge tank to thickeners at 125′ head.



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Impressionistic drawing of the flow in the Centri • Cleaner.

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An installation of Bauer No. 622 Centri • Cleaners (Howard Smith type) handling pulp slurry from knotters. Note that five are hooked up together in the primary group for the first pass. A single one handles the second pass; that is, it reworks the rejects from the primary group. Capacity of this compact assembly of Centri • Cleaners is 250 tons per day.

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Joaquim Jose de Sousa Arocha, Laboratory Chief, Fabrica de Papel de Matrena, Matrena, Portugal, a graduate of Jacome Ratton Industrial and Commercial College.

Elbert H. Barclay, Construction Engineering Estimator, Union Bag & Paper Corp., Savannah, Ga., a 1938 graduate of Lehigh University.

H. Richard Barnett, Manager, Bulk Materials Dept., Manhattan Paste & Glue Co., Inc., Brooklyn, N. Y., a 1924 graduate of Cambridge University.

W. Francis Bathon, Research Dept., Elk Paper Mfg. Co., Childs, Md., a 1949 graduate of Loyola College.

Carl Botkin, Chief Chemist, Charles Bruning Co., Inc., Teterboro, N. J., a 1937 graduate of New York University.

Thomas H. Broderick, Superintendent, Champion International Co., Lawrence, Mass. Attended Harvard University.

Philip D. Caine, Sales Representative, Becco Sales Corp., Boston, Mass., a 1942 graduate of Lowell Textile Institute.

Richard J. Carney, Production Manager, Folding Carton Div., A. George Schulz Co., Milwaukee, Wis. Attended the University of Notre Dame and University of California, Los Angeles.

Burt W. Collins, Chief Chemist, International Paper Co., Southern Kraft Div., Bastrop, La., a 1932 graduate of Alabama Polytechnic Institute.

Douglass M. Compton, Board Mill Superintendent, Robertson Paper Box Co., Inc., Montville, Conn., a 1932 graduate of Yale University.

Joseph E. Cotter, Assistant Manager, Cartonajes Estrella, S. A. Mexico, D. F.

William J. Darmstadt, Sales Engineer, The Babcock & Wilcox Co., New York, N. Y. Attended Rutgers University.

Robert W. Doe, Assistant Superintendent of Pulp Mills, West Virginia Pulp & Paper Co., Mechanicville, N. Y., a 1939 graduate of the University of Maine.

Arthur G. Eltzroth, Laboratory Analyst, The Container Corp. of America, Chicago, Ill., a 1952 graduate of Tri-State College.

Almon M. Fairbrother, Production Manager, The Champion Paper & Fibre Co., Canton, N. C., a 1930 graduate of the New York State College of Forestry.

Bernard Gershill, Cannery Manager, Standard Canners & Packers Ltd., Port Elizabeth, S. A., a 1936 graduate of the University of Cape Town with a Ph.D. degree.

Francisco C. Gonzalez, J. E. Sirrine Co., Greenville, S. C., a 1948 graduate of The Citadel.

William C. Hayes, Paper Mill Superintendent, National Container Corp., Jacksonville, Fla.

Walter B. Hobbs, Jr., Pulp Mill Superintendent, National Container Co., Jacksonville, Fla. Attended the University of Florida.

Raphael F. Illescas, General Director, Laboratorios Nacionales de Fomento Industrial, Mexico, D. F., a 1921 graduate of the School of Chemistry, University of New Mexico.

Vernon C. Iverson, Nekoosa-Edwards Paper Co., Port Edwards, Wis., a 1950 graduate of Luther College.

John M. Johnston, Master Mechanic, Oxford Paper Co., Maine Coated Div., Rumford, Me.

Thomas J. Kavanagh, Salesman, B. F. Perkins & Son, Inc., Holyoke, Mass.

John S. Kay, Assistant to Paper Mill Superintendent, Rhinelander Paper Co., Rhinelander, Wis.

Thomas M. Lalor, Supervisor, Champion Paper & Fiber Co., Pasadena, Tex., a 1937 graduate of Syracuse University.

Edwin E. Lambeck, Chemist, Kalamazoo Vegetable Parchment Co., Kalamazoo, Mich., a 1943 graduate of Western Michigan College.

Robert J. Leahy, Technical Service Representative, Hercules Powder Co., Wilmington, Del., a 1948 graduate of Villanova College.

Kenneth E. Leger, Chemist Westfield River Paper Co., Inc., Russell, Mass., a 1951 graduate of Northeastern University.

Henry L. Levkoff, Manager, L. Sonneborn Sons, Inc., New York, N. Y., a 1942 graduate of Columbia University.

Robert E. Linde, Research Chemist, Crown Zellerbach Corp., Camas, Wash., a 1951 graduate of the University of Minnesota.

George E. Mackin, General Manager, Green Bay Paper & Pulp Co., Green Bay, Wis. Attended the University of Wisconsin.

Edward F. Manogue, Superintendent of Paper Mill, Gibralter Corrugated Paper Co., Inc., North Bergen, N. J.

John O. Markward, Research Engineer, S. D. Warren Co., Cumberland Mills, Me., a 1947 graduate of the University of Texas.

Ernest A. Mateer, Secretary, Canadian Paper Box Mfrs. Assn., Toronto, Ont., Canada.

Paul J. McLaughlin, Research Chemist, Hercules Powder Co., Wilmington, Del., a 1937 graduate of the University of Pennsylvania with a Ph.D. degree in 1943.

John J. Minelli, Quality Control Director, Eureka Specialty Printing Co., Scranton, Pa., a 1931 graduate of Pennsylvania State College.

E. Kenneth Moore, Chemical Engineer, Champion Paper & Fibre Co., Hamilton, Ohio, a 1925 graduate of the University of Cincinnati.

Harold B. Moors, Quality Control Supervision, International Paper Co., Single Service Div., Kalamazoo, Mich., a 1943 graduate of Clarkson College of Technology.

Richard J. Moser, Laboratory Research, Kalamazoo Vegetable Parchment Co., Parchment, Mich., a 1952 graduate of Michigan State College.

Osgood H. Munger, Chemist, Oregon Pulp & Paper Co., Salem, Ore., a 1952 graduate of Oregon State College.

Lamar M. Murray, Sales and Service Representative, Improved Paper Machinery Corp., Nashua, N. H. Attended Louisiana State University.

Y. Nakaba, Chief, The Osaka Kogyo Shikensho, Shikoku Branch, Hanamiyacho, Tamakmatsu, Kagawaken, Japan, a 1930 graduate of Kiriu Technical College.

Carl M. Norton, Jr., Production Chemist, St. Joe Paper Co., Port St. Joe, Fla., a 1948 graduate of Clemson Agricultural & Mechanical College.

Henry A. Olson, Chemist, Premoid Corp., West Springfield, Mass., a 1938 graduate of the University of Minnesota.

John J. Owen, Jr., Engineer, Union Bag & Paper Corp., Savannah, Ga., a 1952 graduate of Louisiana State University.

Cecil C. Parvin, General Superintendent, Nekoosa-Edwards Paper Co., Port Edwards, Wis.

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George E. Prentiss, Technical Assistant to Machine Coated Superintendent, Oxford Paper Co., Rumford, Me., a 1943 graduate of Cornell University.

John F. Richmond, Assistant Manager, The Ace Electrotype Co., Cleveland, Ohio.

Guy E. Sanford, Sales-Chemical Engineer, Goslin-Birmingham Mfg. Co., Inc., Birmingham, Ala., a 1950 graduate of the University of Alabama.

Leo Sokkola, Laboratory Chief, Haarlan Selluloosayhtio, Lievestuore, Finland, a 1927 graduate of the University of Jena, with a Ph.D. degree.

Robert F. Sutherland, Technical Superintendent, South African Board Mills Ltd., Umgeni, Durban, Natal, S. A. Attended Heriot-Watt College.

Charles S. Sweitzer, Mill Technician, Gardner Board & Carton Co., Middletown, Ohio, a 1952 graduate of the New York State College of Forestry.

Heinz Temming, Director, Peter Temming A. G., Gluckstadt bei Hamburg, Germany, a graduate of the Universities of Munchen and Berlin, with a Ph.D. degree.

Rodolfo P. Tenorio, General Manager, Adhesivos Resistol, S. A., Mexico, D. F., a 1924 graduate of Rensselaer Polytechnic Institute.

Juan A. Tettamanti, Manager of Production, Industrias Celulosicas Regionales, S. A., Roasario, Argentina.

Raymond M. Tokarz, The Chesapeake Corp. of Virginia, West Point, Va., a 1943 graduate of Virginia Polytechnic Institute.

John E. Vassie, Technical Director, International Paper Co., Southern Kraft Div., Cullendale, Ark., a 1933 graduate of the University of Florida.

Ivar Vestby, Chief Chemist, Hunsfos Fabrikker, Vikeland, Norway, a 1948 graduate of the Norwegian Institute of Technology.

John C. Waddell, Senior Project Assistant, American Box Board Co., Grand Rapids, Mich., a 1944 graduate of the United States Naval Academy.

Lewis H. White, Jr., Bleach Plant Superintendent, St. Regis Paper Co., Pensacola, Fla., a 1947 graduate of Mississippi State College.

William F. White, Sales Engineer, Oliver United Filters, Inc., Chicago, Ill., a 1948 graduate of Bucknell University.

William A. Welsh, Jr., Chemical Engineer, Container Corp. of America, Wilmington, Del., a 1950 graduate of the University of Delaware.

Edward G. Wilson, Superintendent, Bleached Sulphate Pulp Mill, Kalamazoo Vegetable Parchment Co., Ltd., Espanola, Ont., Canada. Attended Southern California College.

Kenneth R. Wink, Chemical Engineer, Kimberly-Clark Corp., Neenah, Wis., a 1935 graduate of the University of Wisconsin.

Adolf Wolbank, Manager, Papier und Zellulosefabrik, Brigl & Bergmeister A. G., Niklasdorf/Mur., Austria, a 1938 graduate of the University of Vienna.

Donald E. Yost, Engineer, Combined Locks Paper Co., Combined Locks, Wis., a 1941 graduate of Dunwoody Institute.

TAPPI Notes

Charles A. Bassemir, formerly of the Container Corp. of America, is now associated with O. L. West, Agent for Buckman Laboratories, Inc., and Keratene Co., Narberth, Pa.

Maxwell H. Boyce, of the Dyestuffs Div., E. I. du Pont de Nemours & Co., Inc., has been transferred from Chicago to Philadelphia, Pa., as Sales Supervisor.

John E. Bauman, formerly Assistant Manager of the Paper Container Mfg. Co., is now with the Solo Cup Co., Kansas City, Mo.

R. A. Butler, is now Assistant to the Management, Pacific Mills, Inc., Oceans Falls, B. C.

Gardner H. Chidester, Chief of the Pulp and Paper Div., U. S. Forest Products Laboratory, Madison, Wis., is at present spending three months with the Forestry Div. of the United Nations Food & Agriculture Organization in Rome, Italy.

Harold E. Corbin of Socony-Vacuum Oil Co. has been transferred from Brooklyn, N. Y., to Boston, Mass., as a Sales Engineer.

Chester D. Crowell, Jr., formerly student at the New York State College, of Forestry, is now in the Technical Dept. of the Union Bag & Paper Corp., Savannah, Ga. (at present hs is on leave of absence as 2nd Lieut. at Camp Benning, Columbus, Ga.)

Maynard J. Cusack, formerly with the Riegel Paper Corp., is now Vice-President of Shawano Paper Mills, Chicago, Ill.

Robert E. Collins, formerly with the West Virginia Pulp & Paper Co., is now Sales Engineer for the Sprout, Waldron Co., Inc., Muncy, Pa.

Arthur H. Damon, formerly with the News Print Service Bureau, is now a Chemist for the Robert Gair Co., Tonawanda, N. Y.

John E. Donahue of the Hercules Powder Co. has been transferred from Kalamazoo, Mich., to Wilmington, Del., as a Technical Service Engineer.

Francis J. Early, formerly of the Ashland Paper Mills is now Assistant Chemist for A. P. W. Products, Inc., Albany, N. Y.

August S. Erspamer, formerly of P. H. Glatfelter Co., is now General Superintendent of the St. Helens Pulp & Paper Co., St. Helens, Ore.

J. Alden Extrom, formerly of Chas. T. Main, Inc., is now Technical Production Superintendent for the Alton Box Board Co., Alton, Ill.

Donald P. Finnegan, formerly student at the New York State College of Forestry, is now Assistant Test Supervisor for Great Northern Paper Co., Millinocket, Me.

Henry D. Fisher, formerly of the Falls Paper & Power Co., is now Assistant to the Director of Pulp Utilization, Scott Paper Co., Chester, Pa.

Robert W. Gassin, formerly of J. E. Sirrine Co., is now Design Engineer for the Electronic Div., Century Metalcraft Corp., Van Nuys, Calif.

Augustine C. Groom, formerly of the Mead Corp., is now Chemist for the Robert Gair Co., Inc., Research Div., Uncasville, Conn.

Wm. O. Gutzwiller, Chemist of the Howard Paper Mills Inc., has been transferred from Dayton, Ohio, to the Maxwell Paper Co. Div., Franklin, Ohio.

Mozamuel Haque, formerly student at the University of Maine, is now a student in the Pulp and Paper Dept., Western Michigan College, Kalamazoo, Mich.

John M. Hayward, formerly of the Kalamazoo Vegetable Parchment Co., is now Assistant General Manager of the Kalamazoo Label Co., Kalamazoo, Mich.

Russell F. Hazelton, formerly Associate Professor of Chemical Engineering at the North Carolina State College, is now in the Nitrogen Div., Allied Chemical & Dye Corp., Hopewell, Va.

Eleanor F. Horsey, formerly of the Mathieson Chemical Co., is now a Chemical Technologist for the General Service Administration, Washington, D. C.

Jack E. Jayne, formerly Student at The Institute of Paper Chemistry, is now Research Chemist for the Kimberly-Clark Corp., Neenah, Wis.

Royal S. Kellogg, has changed his mail address from Bradenton, Fla., to Palmetto, Fla.

W. Oliver Kincannon, Jr., Technical Service Representative of the Hercules Powder Co., has transferred from Kalamazoo, Mich., to Milwaukee, Wis.

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A. Moltke Leth, formerly of Fabrica El Progresso Industrial, is now Resident Mill Manager for Cia. de las Fabricas de Papel de San Rafael y Amexas, S. A. Mexico, D. F.

Frank J. Lovegren, formerly of W. C. Hamilton & Sons, is now in the New Production Development Dept., P. H. Glatfelter Co., Spring Grove, Pa.

Richard L. Maeder, formerly Chemical Engineer for The Robert Gair Co., Inc., is now with the Donnacona Paper Co., Ltd., Donnacona, P. Q.

Rudolf A. Maronne, formerly of the Star Paper Mills, Saharanpui, India, is now Pulp & Paper Specialist in the Forestry Div., ETAP, United Nations Food & Agriculture Organization, Rome, Italy.

Fred K. Mesek, of Johnson & Johnson has been transferred from Chicago, Ill., to Dallas, Tex., as Technical Director.

Raymond A. Nelson is now Plant Engineer of the Minnesota and Ontario Paper Co., International Falls, Minn.

Samuel R. Olsen of the Champion Paper & Fibre Co. has been transferred from Pasadena, Tex., to New York City.

Henry C. Pan, formerly of the Union Bag & Paper Corp., is now Chemical Engineer for the Chemical Construction Corp., American Cyanamid Co., Linden, N. J.

Milton R. Roberts, formerly of the Hudson Pulp & Paper Co., is now Chief Chemist for the St. Regis Kraft Corp., Jacksonville, Fla.

A. Sensor Ryan, formerly of Reichhold Chemicals, Inc., is now Technologist for the Weyerhaeuser Timber Co., Longview, Wash.

Felix W. Saco, formerly of the Cameron Machine Co., is now Mechanical Development Engineer for the Permutit Co., New York, N. Y.

Robert E. Spearin, formerly student at The Institute of Paper Chemistry, is now with the Combined Locks Paper Co., Combined Locks, Wis.

Conrad Steel, formerly of Hunsfos Fabrikken, Vikeland, Norway, is now Engineer for the Beloit Iron Works, Beloit, Wis.

W. A. Stone of the Mathieson Chemical Corp has transferred from New Orleans, La., to Mobile, Ala., as Technical Sales Representative.

Arnold R. Sundholm, formerly Manager of Wartsita-Cellulose A/B, Aanekoshi, Finland, is now with Amotfors Papersbruks A/B, Amatfors, Sweden.

W. J. Sutton, formerly Executive Director of Bowaters Development and Research, Ltd., London, England, is now with Bowaters Southern Paper Co., Calhoun, Tenn.

Hermann F. J. Wenzl, formerly general Manager of Fedrigoni & Cie Paper Mills, Ltd., Verona, Italy, is now Consultant at Lucerne, Switzerland.

Roland Wilber, formerly of Potlatch Forests, Inc., is now Acting Manager of the Fernstrom Paper Mills, Pomona, Calif.

L. G. Wilkins, formerly of the Oxford Paper Co., is now in the Pulp Div., Weyerhaeuser Timber Co., Longview, Wash.

C. Frederick Young, formerly of the Monadnock Paper Mills, Inc., is now Technical Director of the Hawthorne Paper Co., Kalamazoo, Mich.

Robert N. Zabe, formerly of the Oxford Paper Co., is now in the Research Dept. of Champion Paper & Fibre Co., Hamilton, Ohio.

* * *

Note: The 1953 *TAPPI Year Book* is now in preparation. Members of the Technical Association are urged to check their listings in the 1952 *Year Book* and advise the Secretary of TAPPI, 122 E. 42nd Street, New York 17, N. Y., of any changes that should be made in the new directory.

Industry Notes

Production

The monthly production average for paper and paperboard for the first eight months of 1952 was 1,992,701 tons, more than 11% below the 2,244,184 tons monthly average for the same period in 1951. Production of paperboard in the first six months was a million tons under the production for the first half of 1951. The annual rate of production of paper and paperboard production was just under 24 million tons for the first eight months of this year.

Wood pulp production for the January–July period of 1952 was 9,560,000 tons, a decline of 1% over the comparable 1951 period. Consumption of wood pulp declined 4%. The total consumption was 9,994,000 tons. Imports from Canada and Europe of wood pulp for 1952 through July amounted to 1,042,301 tons, a decline of 29% under imports for the same 1951 period. Imports from Canada of 914,761 tons represented a 17% decline, while imports from Europe of 127,540 tons, a decline of 65%. Exports of wood pulps for the period totaled 149,000 tons, an increase of 30% over the 1951 period.

Total pulpwood receipts for 1952 through July amounted to 15,819,000 cords, a decline of 3% under comparable 1951 receipts. Domestic receipts of 14,508,000 cords showed a 23% decline and imports of 1,311,000 cords were down 9%.

At the end of July, 1952, receipts of wastepaper amounted to 4,368,000 tons, a decline of 28% under receipts for the first seven months of 1951. However, in the last quarter of this year it appears likely that manufacturers of paperboard and roofing materials are likely to consume more wastepaper than in any previous quarter of 1952, but due to softness of the pulp markets, the paper manufacturers may use somewhat less of the high grades or pulp substitutes than during the early part of the year.

Bagasse

Acting under authority of a congressional subcommittee, government scientists under the direction of Dr. R. B. Hobbs, acting chief of the paper section, at the National Bureau of Standards, in Washington, D. C., have investigated the possibilities of making a substitute newsprint from bagasse fiber.

The chief problems of the government's investigators, according to reports, are economic rather than scientific, and practical instead of theoretical.

While the experiments at the National Bureau of Standards were under authority of a congressional subcommittee, it is understood that members of congress from both Louisiana and Florida have favored a similar investigation for some time. According to government reports, the production and availability of sugar cane bagasse from Louisiana in 1950 was 750,000 tons on a bone-dry basis while only 500,000 tons bone dry were produced in 1951. In Florida 125,000 tons bone dry were produced and available in 1950 while only 115,000 tons, on the same basis, were available in 1951. Approximately one third of sugar cane bagasse is pith while two thirds is available fiber.

While both Louisiana and Florida produced less bagasse in 1951 than in 1950, the world production and availability increased from 22,574,000 bone-dry tons in 1950 to 24,793,000 tons in 1951. Countries producing over one million tons bone dry annually are summarized below.

Country	*1950*	*1951*
Hawaii	1,000,000	1,100,000
Puerto Rico	1,300,000	1,400,000
Cuba	6,500,000	8,000,000
India	1,500,000	1,600,000
Brazil	1,800,000	1,900,000
Australia	1,100,000	900,000

A report issued by the Mutual Security Agency, July 16, 1952, prepared by Dr. J. E. Atchison, states, ". . . considering the total quantity (bagasse) available is somewhat misleading because there is a great demand for most of this

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material as fuel for the sugar mills. It has been suggested that other fuels should be substituted for bagasse in the sugar mills so that more valuable products could be produced from this high quality material. In many areas, this is absolutely beyond the realm of economic probability. Many sugar mills are located in areas where fuel imports would be extremely expensive and in these areas the fuel value of the bagasse is of vital importance to the sugar mills. Furthermore, any substitution of other fuels for bagasse would involve large investments on the part of the sugar mills not only for replacement of boiler plant equipment, but for handling and storage of the other fuel.

"Still another factor against use of bagasse in many of these areas is the fact that the sugar mills are all small and scattered and there would be no good means of concentrating the bagasse in one location. Since pulp mill operations must be year-around operations, whereas sugar mill operations are only three to five months per year, the bagasse must be baled, treated to prevent deterioration, and stored for use as needed. A baling station alone requires a large investment and it would be necessary to have such a baling station at each sugar mill from which bagasse is obtained.

"Because of the above facts, as well as other difficulties where only small sugar mill production units are involved, the utilization of sugar cane bagasse for production of pulp and paper is necessarily limited to certain areas. These areas must have large sugar mill production units. This is particularly true if merely surplus bagasse is to be used.

"If other fuel is to be substituted for bagasse in the sugar mills, then the area must have access to low fuel supplies such as oil, coal, or natural gas. Even when extremely low cost fuel is available, sugar cane bagasse is not a cheap raw material for the production of pulp and paper. Louisiana probably has the most favorable conditions in this respect of any sugar-producing area in the world. In Louisiana natural gas is now selling for $0.17 per 1000 cu. ft., and it requires 12,000,000 cu. ft. to replace one ton of dry bagasse as fuel. Thus the fuel replacement value of the bagasse is only slightly more than $2.00 per dry ton. However, it has been estimated by several reliable engineers, including Dr. Arthur G. Keller, Professor of Chemical Engineering at Louisiana State University, that bagasse delivered to the pulp mill would cost $10 to $12 per dry ton. This means that the actual total cost of the bagasse on the dry basis to the pulp mill is some $8 to $10 higher than its mere replacement value as fuel. With these facts in mind, it is not difficult to understand the difficulties which bagasse pulp and paper mills will have in high-cost fuel areas. At best it will be difficult for them to compete with mills using southern pine wood at $14 per cord, unless they produce a product for which bagasse fiber is especially suited."

The experimental pulp and papermaking studies made at the National Bureau of Standards were reportedly conducted from whole raw bagasse received at the Bureau from Louisiana.

On August 8, H. B. McCoy, Director of the Office of Industry and Commerce, in the U. S. Department of Commerce, welcomed leading newspaper executives to Washington to observe the results of the Bureau's experiments. It is reported that the publishers' subjective judgment of the samples presented to them were as important as the scientific measurements available. They were asked to consider the samples on the basis of pliability, opacity, printability, and over-all acceptability. The newspaper executives participating at the meeting included P. H. Rice, Augusta, Ga., *Chronicle-Herald;* R. B. Scudder, Newark, N. J., *News;* J. L. Knight, Miami *Herald;* Herman Frame, New York *Herald Tribune;* Wm. Baumrucker, Washington *Times-Herald;* John Cusik, Philadelphia *Inquirer;* Glen James, Centerville, Md., *Record-Observer;* Donald Boyce, Hearst Publications; T. A. Corcoran and Lawton Dix, Louisville *Courier-Journal;* and, Henry Botthof, of the Baltimore *Sun.*

Some of the samples shown closely resembled newsprint paper in appearance, but most were characteristically dirty and poorly formed. These defects, from a papermaker's viewpoint, might have been due to a limitation of equipment at the Bureau; the quality of the raw bagasse; or, to other factors. According to reports the processes tested included the Valite, Kinsley, Horn, da la Roza, and two processes developed at the Northwestern Regional Laboratories in Peoria by Dr. E. C. Lathrop and Dr. Sam Aronovsky. They also included bleached and unbleached and pith and de-pithed fiber.

The results of the work performed at the National Bureau of Standards will soon be presented in a report by the Department of Commerce to the House Judiciary Subcommittee on the Study of Monopoly Power. Rep. Celler, chairman of the subcommittee, plans to hold open hearings some time in September on the Department of Commerce's conclusions according to the *Journal of Commerce.* (*News Print Service Bureau Bull. 416.*)

Consolidated

Another step was taken here yesterday toward using spent sulphite liquor as industrial fuel instead of flowing it into the Fox River when Interlake Mill of Consolidated Water Power & Paper Co. installed a huge new storage tank.

Fabricated at Marshfield, Wis., the 14,000-gal. stainless steel unit was delivered on a heavy-duty trailer which had to follow a special route laid out to miss all low underpasses and weak bridges on the 105-mile trip to Appleton. The tank was unloaded by crane when it arrived and was set on a concrete and steel foundation outside the mill boiler house.

It will be used for storing sulphite liquor which has been concentrated to about six times its original strength in the new evaporator plant currently being installed at Interlake Mill. Concentrated liquor will flow from this tank to the liquor burning furnaces and will replace other fuel now used. Burning the liquor will substantially reduce sulphite pollution of the Fox River all the way downstream to Green Bay.

Nekoosa-Edwards

Spent sulphite liquor previously flowing into the Wisconsin River will be pumped into ponds or lagoons that now are being constructed by Nekoosa-Edwards Paper Co. under the supervision of state officials. Design and specific area for testing were chosen in consultation at the site between company engineers and personnel of the State Committee on Water Pollution headed by State Pollution Commissioner Theodore F. Wisniewski.

Public officials and the mill's technical men recognize that this is an experiment, but they are hopeful it will yield valuable data. Preliminary tests using smaller ponds indicate that this disposal process may improve stream conditions.

Nekoosa-Edwards has built on an island 2,000 ft. from its sulphite pulp mill a lagoon system that consists of two 2000-sq. ft. primary receiving pits and a 4000 sq. ft. secondary pit, all three pits 4 ft. deep and interconnected so that any combination of these may be used at any time. Other parts of the project are a collection system to gather spent liquor from the blow pits, two 70,000-gal. wood stave storage tanks, a pump capable of delivering 140 g.p.m., and 2000 linear feet of 4-in. welded stainless steel pipe with accompanying valves and fittings. Completion of construction depends upon time required for obtaining the stainless steel pipe. Present delivery promises from suppliers point to installation and start-up during November, 1952.

Preliminary experiments with smaller lagoons showed that spent sulphite liquor soaks into the sandy soil of the island at a rate between 20 and 30 gal. per sq. ft. per hr. Daily tests will be made on river water at several sampling stations to detect any seepage of spent sulphite liquor which may occur

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and to measure improvement in stream conditions resulting from this installation.

Southern Pulpwood

Pulpwood production in the South in 1951 reached an all-time high of 14 million cords, or 56% of all pulpwood cut in the nation. The harvest was 1.6 million cords larger than in 1950. It was comprised of 12,442,000 cords of pine, 1,451,000 cords of hardwood, and 168,000 cords of dead chestnut. The region's pulpwood harvest has nearly doubled in the last decade, and now accounts for 18% of the total volume of wood taken from southern forests. The outlook is for still further expansion, since proposed new mills or enlargement of existing ones will soon increase wood requirements about 6 million cords.

Bowaters Southern

Contracts have been let for the construction of a new 50 million dollar newsprint and sulphate pulp mill at Calhoun, Tenn., for Bowaters Southern Paper Corp. The contracts were awarded to three contractors—Fraser, Brace & Co., and Turner Construction Co. of New York and Roane Anderson of Calhoun.

The new mill will be capable of producing 125,000 tons of newsprint and 50,000 of sulphate pulp annually. It is expected to be in operation in 1954. The mill is on the Hiwassee River near Calhoun, between Chattanooga and Knoxville.

Sulphur

The estimated consumption of sulphur for 1953 is 6,637,000 long tons; the total supply, 7,000,000 long tons with an indicated inventory increase of 363,000 tons.

Casein

On October 1 import quotas on casein were increased 15%, raising the allowable yearly imports for July 1, 1952, to June 30, 1953, to 46,000,000 lb., from a previous 40,000,000 lb. quota.

U. S.-Japanese Pulp Mill

The Daily Stock Reporter advises that the first of a series of meetings and discussions has been held in Washington between a group of Japanese forestry experts and U. S. Government officials in the State, Interior, and Labor Depts. on forming a joint American-Japanese pulp mill in Alaska. The pulp would be shipped to Japan for use in her textile and paper mills.

Finland

Finland completed its payments of reparations to Russia on September 19. The list of war reparations consisted of 240 separate items, the largest of which was 573 ships worth 80 million dollars. Next largest was 626,230 tons of paper, cardboard, and cellulose, worth 35 million. This was followed by 22 million dollars worth of lumber, plywood, pulpwood, and thread spools. The reparations included 17 million dollars worth of paper machinery.

Norway

The export tax on wood pulp and paper has been abolished by Norway, retroactive to June, 1952. The removal of the tax was in line with the Norwegian Government's promise of eliminating it once market prices of wood pulp reached a nearer normal level and the tax was no longer necessary.

Saskatchewan

Pulp manufacturing interests from Eastern Canada, the United States, Great Britain, and Switzerland have been investigating the pulp-making possibilities of the Province, where preliminary forest survey data indicate the presence of 24 million cords of standing softwood timber available

The Provincial Government has initiated a program to assist interested companies in establishing pulp and paper mills in the Province.

Tennessee

The Southern Forest Experiment Station, 704 Lowich Bldg., 2026 St. Charles Ave., New Orleans, La., has issued a Forest Survey of Tennessee, copies of which are available from the Station.

Plant Maintenance Show

The Plant Maintenance Conference and Show will be held at the Public Auditorium, Cleveland, Ohio, on Jan. 19–22, 1953. Eight section conferences and 21 round tables will be held. The pulp and paper conference will feature papers on "Maintenance in Paper Mills" by P. W. Sarle, Oxford Paper Co., Rumford, Me., and "Maintenance for Paper Products Plants" by George E. Ellis, Scott Paper Co., Chester, Pa. Details about all programs may be obtained from Banner & Greif, 250 W. 57th St., New York 19, N. Y.

Semichemical Research

For the last six months the Virginia Polytechnic Institute and Louisiana State University projects of the National Council for Stream Improvement, Inc., New York, N. Y., have cooperated on semichemical pulping waste handling. Research has been directed toward finding more economical means of evaporating or concentrating the spent liquor from the neutral sulphite process. The three processes investigated were submerged combustion evaporation, vapor compression evaporation, and selective solvent concentration. It was found that the capital cost for submerged combustion evaporation was much lower than that for conventional evaporators, but that fuel requirements were substantially higher and limited to gas for fuel. Vapor compression evaporation operated with considerably higher efficiency, but was much higher in equipment cost than other methods. Selective solvent concentration employing acetone was found to be costly due to the amount of acetone which had to be separated by distillation for recycling.

Experiments were also conducted at Virginia Polytechnic Institute with the heat hydrolysis process. It was found to be less effective on semichemical liquor than on acid sulphite. This is due to the fact that the former contains relatively little hexose sugar and is neutral in reaction.

Reports covering these studies have been prepared and will be published shortly.

Penobscot Chemical Fibre

Walter V. Wentworth, who has been Manager of Mills for the Penobscot Chemical Fibre Co. at Great Works, Me., for 54 years, was elected a Vice-President of the company. Lynwood S. Hatch, who has been Mr. Wentworth's assistant for the past ten years, has succeeded him as Manager of Mills.

Fiber Fabric Group

The Fiber Fabric Group of the Tissue Association, Inc., 122 E. 42nd St., New York, N. Y., has issued an interesting booklet, telling the story of the resurgence of the fiber fabric industry. Samples of fiber fabric are used as part of the booklet covers. Photographs show the various stages of fiber fabric manufacture.

Lifeblood

Abitibi Power & Paper Co., Ltd., Toronto, Ont., has issued an interesting 20-page pamphlet entitled "Lifeblood—A Story of the Forest of Abitibi."

Tannin

"Tannin from Waste Bark," Bulletin No. 39 of the Northeastern Wood Utilization Council, Box 1577, New Haven 6,

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Conn., has been issued as a 31-page pamphlet. Included are articles on "Operation of a Tannin Extract Plant" and "Potential Tannin Supplies from Domestic Barks." There is an interesting chapter on "Methods of Bark Separation (Air-Flotation, Thermo-Pressure, Cyclone, and Portable Debarkers)." Copies are available from the Council at $2.50 per copy.

ASTM

Robert J. Painter has been elected Executive Secretary of the American Society for Testing Materials, Philadelphia, Pa. Raymond E. Hess was elected Associate Executive Secretary and Editor in Chief of Publications.

Storage of Lumber

The Forest Products Laboratory, Madison, Wis., has issued a useful report on "Storage and Handling of Lumber" (No. D1919).

Paper Production Capacity

In the 40 years between 1889 and 1939 American paper production averaged 76.3% capacity. High was 82.9% in 1917; low was 58.3% in 1932. From 1940 to 1951 the average was 92.4% with a low of 85.6% in 1945 to a high of 103.3% in 1951.

Frozen Foods

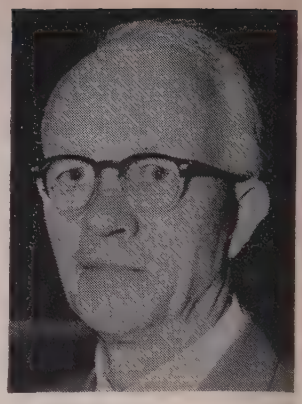
The combined frozen food pack for 1952 is expected to reach 3.2 billion pounds. In 1951 it was 2.5 billion pounds.

Forestry Degrees

During 1950–51, a total of 1730 persons, including eight women, received B. S. degrees in forestry from American colleges. In addition 260 received M.S. degrees and 17 received Ph.D degrees.

Institute of Paper Chemistry

Roland E. Kremers, until recently connected with the Central Laboratories Div. of General Foods, Inc., has been appointed Research Associate in Organic Chemistry at The Institute of Paper Chemistry, Appleton, Wis.



R. E. Kremers, The Institute of Paper Chemistry

Gair

George J. Tooker has been appointed Division Manager of Robert Gair's Natick (Mass.) Boxboards Div.

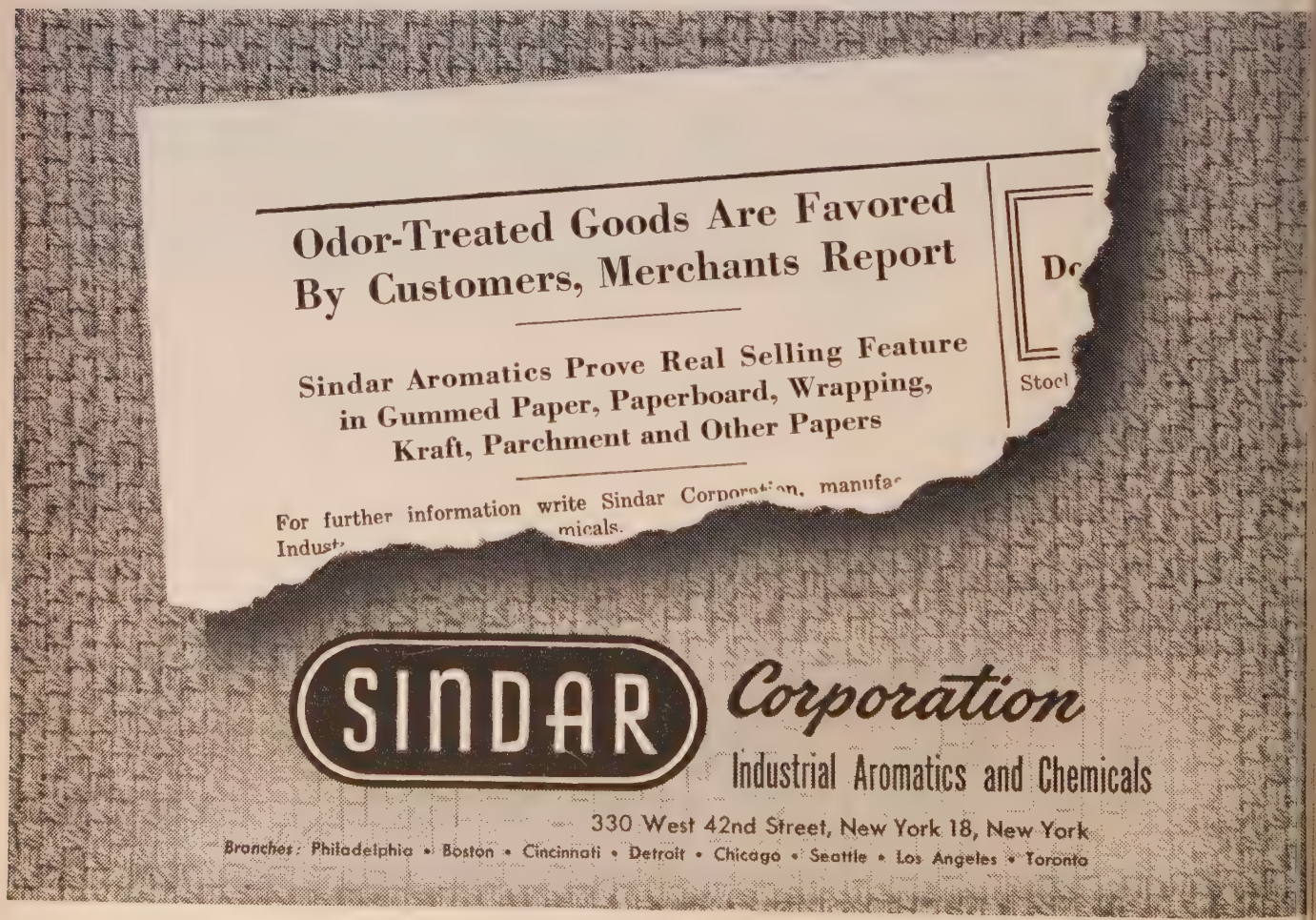
Russell D. Scribner has been appointed Division Manager of its Thames River (New London, Conn.) mill and box shop.

Ball Bros.

Burnham B. Holmes has been appointed General Manager of Paper, Purchasing, and Traffic of Ball Brothers Co., Muncie, Ind. George H. McGowan, formerly of the Anchor Hocking Glass Corp., has been appointed Manager of the Paper Products Div.

University of Maine

The University of Maine has started a new course in Paper Mill Management. It will deal with industrial methods of managing the various operating departments of a paper mill and the functions of those departments. The course will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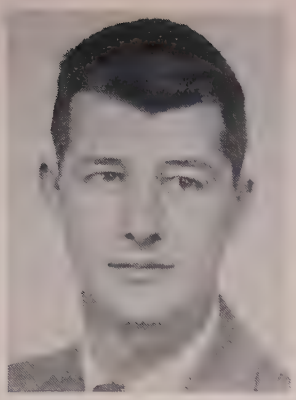

A. S. Campbell, University of Maine

be taught by Charles M. Howell, formerly of the Scott Paper Co.

Dean Ashley S. Campbell of the College of Technology at the University of Maine has announced the Pulp and Paper Foundation program to include all departments of the College of Technology and the Forestry Dept. Under this program, tuition scholarships will be available to qualified junior and senior engineering and forestry students who plan to enter the pulp and paper and allied industries.

Recipients of recent Foundation grants and the companies by whom they are employed are as follows: Donald R. MacKinnon, Oxford Paper Co.; Robert N. Zabe, Champion Paper & Fibre Co.; Roy P. Webber II, Air Force Service; Dwight D. Frye, Union Bag & Paper Co.; James E. Ayer, West Virginia Pulp and Paper Co.; Frank A. Butler, Chemical Corps, Army Service; John M. McClure, S. D. Warren Co.; William E. Pomeroy, Robert E. Perry, Scott Paper Co. Additional recipients are Eugene Cunningham, '53, Carl D. Perkins, '54, and Robert Touchette, '54.

Transportation and Packing Survey

In an 88-page final report published in Washington, D. C., this week, the Transportation & Packing Survey presented the results of three years' investigation into the causes of damage to goods shipped by rail in fiber boxes. Based on its work, the report suggests that the railroads could reduce their loss and damage payments from the present 100-million-dollar-a-year level by setting up a new bureau which would locate and work with companies whose products are damaged excessively during rail shipment. By concentrating its efforts on shippers with the highest claim records, this bureau would supply advice and assistance where it is most needed and where the greatest reduction in loss and damage claims can be made.

The Transportation and Packing Survey was organized jointly by the railroads of the United States and the Fibre Box Association in 1948 when loss and damage payments had reached an all-time high of 135 million dollars and threatened to go higher in 1949. At the time, basic information was lacking on which an effective program for reducing these claims could be based. The survey was set up to provide both a broad and a detailed picture of the factors which produce damage to goods shipped in fiber boxes, to find the commodities which are most susceptible to damage, and to determine whether some shippers of a given commodity consistently incurred more damage than other manufacturers of the same commodity. During the three years in which it operated, the survey inspected 3440 carloads of products from more than 1000 different manufacturers. The results of these inspections showed that less than 10% of the cars had sustained over 60% of the damage. It was because of this finding that the recommendation was made for the

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establishment of a bureau to work with individual companies on their carloading problems. One wine company on the West Coast sustained an average claim of $165 on each carload while another winery had an average claim of only $4.00 on each car.

The three most prominent causes of damage found were: rough handling of the car by the railroads, which was responsible for 27% of all the damage; poor arrangement of the load by the shipper, which caused 19%; and loose loading of the boxes in the car, which produced 16% of the claims. The remainder of the damage was divided among 18 other minor causes.

It was reported that, among those products covered in the survey, wine in gallon jugs and juices in 46-oz. cans were most susceptible to damage. On carload shipments of gallon jugs of wine, the average claim was $227 per car, and on 46-oz. cans of juice the average claim was $70 per car. This is in contrast to an average claim of $1.00 per car on beer in cans.

The report also presented data on a number of other factors which have an effect on claims, such as the distance traveled, the weight of the load, the type doorway protection used, the condition and age of the car, the manner of stowage, the closure and fit of the box, and the type protection placed on the floors and floor racks.

The Transportation and Packing Survey is located at 1415 K St., N. W., Washington 5, D. C.

Forest Fires

Forest fire losses in the United States were the lowest in history, statistics recently released by the U. S. Forest Service show. (USFS figures include Hawaii.)

The report lists 164,090 fires that burned 10,781,039 acres of forest land during 1951. The previous low in amount of damage was in 1949 when 193,774 fires burned over 15,397,419 acres. In 1950, 208,402 fires burned 15,518,540 acres.

Of the 639,540,000 acres needing protection in the United States, only 63,624,000 are without it. Although 65% of the fires last year occurred on the protected land, only 33% of the area burned—3,526,373 acres—was on these protected acres.

Once again in 1951 incendiarists caused the greatest number of fires. Of the 105,868 fires on protected lands, 39,396 were maliciously or deliberately set. "Firebugs" in the eleven southern states started 36,259 of the incendiary fires. Debris burners caused 19,590 fires in the United States, while smokers were to blame for 18,390.

The eleven southern states showed a fire loss of 8,702,926 acres burned. Florida with a toll of 3,561,388 acres burned led the nation in fire losses. Nearly one third of the area burned in the entire country is in this one state. A total of 3,066,000 acres was burned in Florida's area without organized protection.

Burning conditions generally were considered "severe" over most of the western United States during 1951 and "normal to favorable" throughout the remainder of the country. The Lake States region had one of its best years. All the central states, except Missouri, had a good season in 1951.

Losses on federally owned land were higher in 1951 than in 1950.

Gould

Gould Paper Sales Corp. has recently established a New York City office at 122 E. 42nd St., with E. J. Doonan as Manager of Sales. This corporation is the distributing company for the products of Gould Paper Co. of Lyons Falls, N. Y., in the groundwood specialty field. It also exports these and other grades into the world market.

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"Continuous Beater & Refiner"

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INSTALLATIONS: SULPHITE-KRAFT PULPS, (BL. & UNBL); BLEACHED SODA PULP

Fifteen years ago a small Kraft Pulp Mill used a No. 1 Claflin Refiner on washed pulp ahead of screening. Washed pulp to Claflin to screen with accepted stock to wet machine chest and screen rejects back to digester and above cycle. Result, pulp required less time and power to repulp, beat and process than with other pulps.

SPECIFIC OPERATING INFORMATION FURNISHED UPON REQUEST.

SALES AGENT, U. S. & CANADA: **THE EMERSON MFG. CO.,** LAWRENCE, MASS.

THE HERMANN MFG., CO. LANCASTER, OHIO

V. V. Vallandigham, Potlatch Forests, Inc.

Potlatch

William P. Davis, President of Potlatch Forests, Inc., Lewiston, Idaho, has announced the appointment of Vance V. Vallandigham as Manager of Research and Development for Potlatch's paper division. In his new position Mr. Vallandigham will be concerned with new developments, technical services, and will coordinate production and quality control problems. For the time being he will continue his residence in Chicago and will divide his time between the plant at Lewiston and the company's customers.

Potlatch Forests, Inc., which started its new paperboard mill about one and a half years ago, manufactures solid bleach sulphate board. The mill specializes in the production of food container board of all kinds, including milk bottle stock, butter carton stock, cup stock, ice cream container board, meat boards, etc.

Prior to taking up his new duties with Potlatch on August 1, Mr. Vallandigham was Technical Director of the paper department of the Kelco Co.

Soundview

Scott Paper Co.'s Soundview Div. at Everett, Wash., has recently installed Black-Clawson drives on the wet ends of their No. 1 and No. 2 pulp drying machines.

These Black-Clawson No. 1 spiral-bevel gear drives, eight in the single shipment, are installed on two of the four Fourdrinier pulp drying machines at the Soundview mill.

Four drives were installed on No. 1 machine; at the couch, first, second, and third presses. Four drives were installed at the same positions on No. 2 machine.

The drives were furnished complete with drive-in shafts and flexible couplings. Each drive is equipped with a Fawick Airflex clutch to provide for control of the drive from the front side of the machine.

The Soundview Div., a recent addition to the Scott Paper Co.'s family of mills across the country, employs approximately 525 persons, maintains its own woodlands, and produces over 600 tons per day of bleached sulphite pulp which is converted into high-grade tissue and paper products at other Scott mills.

Kraft Products Export

Kraft Products Manufacturers Export Association has filed papers with the Federal Trade Commission under the Export Trade Act (Webb-Pomerene Law) as an export trade association for manufacturers of kraft paper, tall oil, and tall oil derivatives. Members of the Association are:

Albemarle Paper Mfg. Co.
Arizona Chemical Co.
Camp Manufacturing Co., Inc.
International Paper Co.
St. Regis Paper Co.
Union Bag & Paper Corp.
Gilman Paper Co.
Hudson Pulp & Paper Co.
Hollingsworth & Whitney

Offices of the Association will be located at 521 Fifth Ave., New York City, in the office of Israel B. Oseas, Counsel to the Association. Association officers are: W. C. Shorter, Camp Manufacturing Co., Inc., President; Leonard Doyle,

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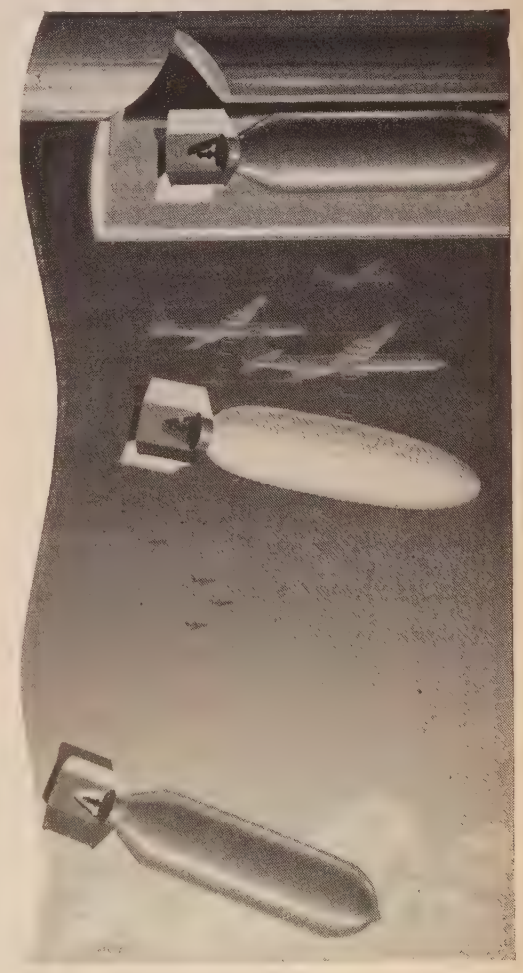

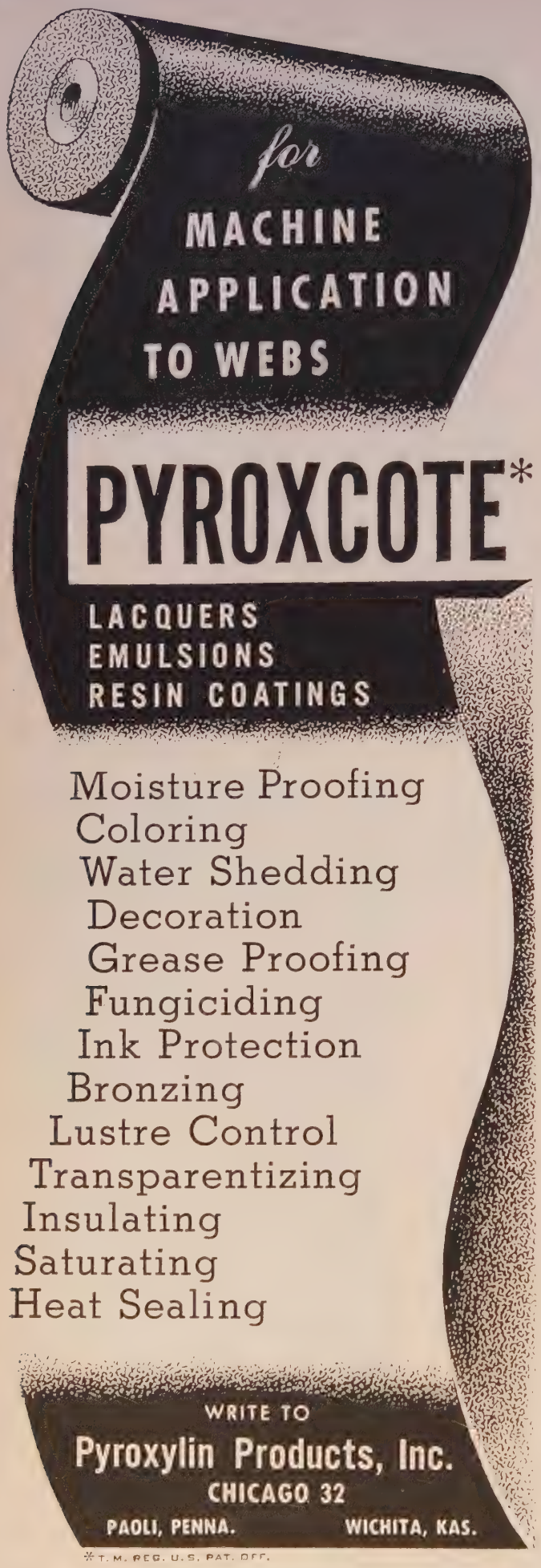

Union Bag & Paper Corp., Vice-President; Israel B. Oseas, Secretary-Treasurer.

New England Pulp and Paper

During the early years of the twentieth century, New England maintained a dominant position in the pulp and paper industry. Since 1930, however, New England's economic position in this industry has slowly declined until it now constitutes about 15% of the entire United States paper industry. Other regions, especially the Far West, have more plentiful supplies of pulpwood of excellent quality. However, it should be noted that recent development of a process for utilizing hardwood will substantially increase New England's raw material supply. Pulp and paper mills in the West and South are run on a much larger scale and have more up-to-date equipment, such as high-speed machinery, resulting in more efficient, low-cost production. In some areas, also, the availability of cheap power is a decided advantage. An expansion of pulp manufacture in New England is limited as well by a lack of available sites on rivers in proximity to wood supplies. New England enjoys a definite advantage in paper manufacture, however, in being close to markets. The region's consumption of paper products is very substantial, and nearby New York and Mid-Atlantic markets are within easy shipping distance.

There are still many opportunities for further industrial development within the pulp and paper industry. One of the most favorable opportunities lies in increasing production of high-grade paper and specialty papers. Such products as writing paper, reinforced paper, wrapping papers, paper towels, ribbons, tracing and thin papers, tablets and pads, ledger paper, lamp shade papers, and book paper are well suited to New England from the standpoint of its technological experience, skilled labor, and available raw materials. A second type of opportunity lies in the manufacture of products based largely on technological developments, such as diapers and diaper linings, printing plates, laminated building papers and food wrappers. Resin-bonding, synthetic and natural rubbers and new coatings applied to various materials suggest many new uses for paper products for both the consumer and industrial markets.

A new type of pulp-molding equipment is being produced by a Maine manufacturer. Although this process is still in the developmental stage, it will, when completed, make available low-cost, custom-molded packages offering maximum protection against impact. Such packaging may find a large market among industrial areas in New England in the electronics and machinery fields. The process utilizes reclaimed paper which is available in ample supply in New England.

Weyerhaeuser

At the Springfield, Ore., mill of the Weyerhaeuser Timber Co. they've engineered out the offensive odors that had been escaping from pulping ("cooking") liquors—something that kraft mill operators have been trying to do for a long time. And they've saved a good deal of sulphur to boot.

They do it by oxidizing (in a large tower) the volatile and odorous sulphides in spent cooking liquor to more stable thiosulphates.

Almost two years ago, the engineers at Springfield put the full-sized tower into operation; it has worked successfully ever since.

But here's the story as it was told to us by Weyerhaeuser's Joseph C. Brown, Technical Director of the Springfield mill, and by H. W. Bialkowsky, Research Director, and Gerrit G. Dehaas, Research Chemist, both of the Pulp Div. of the company.

Whence the Odor. In manufacturing wood pulp by the sulphate (kraft) process, a mixture of sodium hydroxide and sodium sulphate is the active pulping agent. (It's the sodium sulphide that leads to the formation of noisome, volatile su-

phur compounds; these are released from various points in the liquor recovery system.)

During normal operations, the strength and volume of the cooking liquor are kept up by adding makeup sodium sulphate (salt cake) to the spent cooking liquor. This is added in the recovery operation after evaporation and just prior to burning in the recovery furnace. The furnace maintains reducing conditions in the char bed and the sodium sulphate is reduced to sulphide.

However, in a well-operated plant, losses of sodium compounds may be so low that the amount of makeup salt cake needed to maintain liquor inventory may not be enough to maintain "sulphidity" (ratio of sodium sulphide to alkali; 15 to 25% is desirable). When this happens, many mills add elemental sulphur to the system. The recovery furnace reduces the sulphur to sodium sulphide, thus replacing the volatilized sulphur compounds that have been lost.

Simple Air Oxidation. Knowing that these volatile sulphur compounds (mostly H_2S) could be oxidized to more stable compounds by bubbling air into the liquor, Weyerhaeuser researchers set out to learn just how much air was needed. They also wanted to find out if they could skirt the problem of foaming of the liquor, a complication that previous researchers had run into.

After exhaustive laboratory experiments they found that the best bet seemed to be the use of some form of tower to carry out the oxidation.

The laboratory results were engineered through the pilot plant and in October, 1950, the full-sized tower was put on stream.

The tower is designed to handle 300 gal. of black liquor per minute, with an incoming sulphide concentration of 3 g.p.l. Dimensions are: 10 ft. in diameter, 30 ft. high. It's packed with 22 ft. of 4-in. cross partition rings, giving an effective surface of 50,000 sq. ft.

The black liquor is pumped from a storage tank to a distributing arrangement on the tile at the top of the tower. Air is forced into the tower at the top and passes out a vent line from a sump at the bottom. The black liquor overflows the sump, into a secondary sump and from there it is returned to a second storage tank.

They use about 10 cu. ft. of air per minute for each gallon per minute of liquor flow. They ran into no serious foaming troubles and no hydrogen sulphide was found in the air leaving the oxidation tower.

Although the chemical reactions in the tower are complex, the chief reaction seems to be the conversion of sodium sulphide in the spent cooking liquor (or black liquor) to sodium thiosulphate or some other stable sulphur compound. Actually, a water solution of sodium sulphide is relatively stable to air oxidation. But the black liquor is rich in organic compounds and it's believed that this material acts as a catalyst to promote the reaction between sodium sulphide and oxygen.

The sulphide is completely oxidized when the tower load does not exceed 0.67 lb. sulphide ion (expressed as sulphur) per 10,000 sq. ft. of packing surface per minute. Air flows are 4 to 6 cu. ft. per gallon of liquor for normal concentrations of sulphide.

Liquor distribution in the tower permit good wetting with a minimum of splashing. Liquor and air are passed through the tower in the same direction and flows are adjusted to keep regurgitation down. Treated liquor collected in a sump at the bottom of the tower overflows to a secondary sump. The levels in these sumps are carefully adjusted so that the flow of the liquid breaks and stabilizes the foam at the liquid surface. If these liquid levels are not properly balanced very large amounts of foam can be produced.

Before installing the oxidation tower system, operators had to add up to 20 lb. of sulphur per ton of pulp in order to maintain a sulphidity of 14 to 16%. The company estimates

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If you have to run this race in your mill, squirting lubricant on bearing surfaces once an hour, or once in two hours . . . forget it from now on!

USE THE OIL THAT *Clings*

When you lubricate with Magnus KLING-OIL, it stays put so much longer that you can cut the frequency of oiling down to twice a day . . . and even less! KLING-OIL is a safe and sure lubricant, with an SAE viscosity of 30. It is different because, although mobile, it is also "tacky" . . . and clings where it is applied. Where ordinary lubricating oils drip and run off, KLING-OIL stays put . . . providing perfect lubrication for much longer periods.

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When you call off the "Oil-Sprint Handicap" you'll also be surprised to note by your wattmeter that the machines lubricated with KLING-OIL are not pulling as much power . . . even though you are lubricating them less frequently. You'll have a cleaner mill, too, because KLING-OIL doesn't drip and run to make ugly and hazardous puddles under machines. Hundreds of mills use KLING-OIL. *Give it a trial yourself!*

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Butterworth
CALENDER ROLLS

that at the present level of production these additions would amount to nearly 1000 tons per year of critical brimstone. With the tower operating, they've stopped adding sulphur and the sulphidity of the liquor has increased to 21 to 25% with substantially the same amount of makeup salt cake used (about 100 lb. per ton of pulp).

Joseph C. Brown points out that the principle of liquor oxidation is readily adaptable to any kraft black liquor of comparable sulphidity. Weyerhaeuser oxidizes the liquor from Douglas-fir which is their primary wood supply.

The company points out that while generation of H_2S in the evaporating process has virtually been eliminated, the problem is not entirely erased. Hydrogen sulphide is still generated in the recovery furnace and volatile organic sulphur compounds are released in venting the digesters and the multiple-effect vacuum evaporators.

Alexander Mitscherlich Medal

Dr. Emil Heuser, Research Associate Emeritus of The Institute of Paper Chemistry who now lives in La Jolla, Calif., was signally honored recently when he was eulogized by an entire issue of *Das Papier*, the publication of the German Association of Pulp and Paper Chemists and Engineers. The occasion for this anniversary number was Dr. Heuser's 70th birthday, at which time he was also given the Association's highest award, the Alexander Mitscherlick Denkmünze, a coveted commemorative medal.

Emil Heuser

The German publication is unique. It contains a biographical sketch by Professor George Jayme of the Technische Hochschule at Darmstadt where Heuser himself taught for many years and where he inspired scores of students. Many of these who are now prominent names in the cellulose industries contributed scientific articles for this issue. Furthermore, the number contains a complete bibliography of Heuser's publications and patents, of the journals he has edited, and the articles he has published.

Heuser was educated at Munich, Graz, and Karlsruhe where he received the degree of Doktor Ingenieur in 1909. In early life he gained first-hand experience in pulp and paper mills in Germany and Austria. At the Technical University at Darmstadt he served as Professor of Cellulose Chemistry from 1912 until 1923 with an interlude of two years. After the publication of his monograph Lehrbuch des Cellulosechemie, he was Honorary Professor of Cellulose Chemistry at the Technical University of Charlottenburg. In 1926 he became Director of Research of the Canadian International Paper Co.

His appointment as group leader of cellulose chemistry at the Institute came in 1938 and he was made Research Associate Emeritus in 1947. In the following year, Dr. Heuser was the recipient of the TAPPI medal.

Curlator

The Curlator Corp. has moved from 565 Blossom Rd., Rochester, N. Y., to East Rochester, N. Y. Curlator Corp. has appointed Homad Services Ltd., Montreal, P. Q., to represent the company in Canada.

Allis-Chalmers

J. D. Greensward has been named Vice-President and Director of Manufacturing of the General Machinery Div. of the Allis-Chalmers Mfg. Co., Milwaukee, Wis.

P. F. Bauer succeeds Mr. Greensward as the General Manager of the Norwood, Ohio, works of the company.

Allis-Chalmers has issued a four-page bulletin "Allis-Chalmers Frame-Type Centrifugal Pumps" (52 B6691A). Copies are available from the company, 829 S. 70th St., Milwaukee, Wis.

JOHNS-MANVILLE

William J. Bucklee has been appointed General Sales Manager of the Johns-Manville Celite Div. Mr. Bucklee succeeds William D. Van Arnam who has retired.

ANHEUSER-BUSCH

Arthur E. Webber is now General Manager of the Yeast, Malt, and Corn Products Div. of Anheuser-Busch, Inc., St. Louis, Mo. Arthur C. Mohr, formerly Sales Manager of the Corn Products Dept. has assumed the position of Sales Manager of the Specialty Products Div. of the Seymour Packing Co. of Topeka, Kan.

HAROLD E. INGALLS

Harold E. Ingalls is now associated with Ruel E. Taylor, Jr., at 25 Commercial St., Portland, Me., as a manufacturers agent representing Nichols Engineering & Research Corp., Buffalo Pumps, Inc., Detjen Corp., W. B. Coleman, Fabri-Valve Mfg. Co., and Sveen-Pederson Corp. Mr. Ingalls was formerly with Nichols, the International Paper Co., P. H. Glatfelter Co., National Container Corp., and the Continental Paper Co.

Harold E. Ingalls, Manufacturers' agent

WESTINGHOUSE

Westinghouse Electric Corp. will expand its manufacturing operations in the Boston area soon with a $1,250,000 building project at its Sturtwart Div. plant in Hyde Park, Mass. This plant will be devoted to the manufacture of air conditioning equipment, commercial fans, and blowers.

Westinghouse has issued a 50-page book (B-5299) on "Electrical Equipment for Lumber Mills." Copies may be obtained from the company, Box 2099, Pittsburgh, 30, Pa.

F. R. Benedict of Westinghouse gave an unusually interesting address at the recent meeting of the AIEE in Chicago on Electric Power in Industry. Copies may be obtained on request.

HOOKER

Hooker Electrochemical Co. has established a subsidiary, Hooker Chemicals, Ltd., with a plant to be located at Burraard Inlet, near Vancouver, B. C. R. L. Murray will be President and B. Klaussen, Vice-President.

B. & W.

The Babcock & Wilcox Co. has purchased the facilities of a former shipyard from the Brunswick, Ga., Port Authority that will be converted into a plant to build boilers and related equipment. This is the fourth new plant to be announced by the company in the past year. The others being at Wilmington, N. C., West Point, Miss., and Paris, Tex.

G.E.

E. H. Powell has been appointed Assistant Manager of General Electric's Richmond, Va., Apparatus Sales Office. He is succeeded as the Manager of the Trenton, N. J., Apparatus Sales Office by L. S. Brumgard.

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Any pulp slurry may easily be defoamed with a pinch of Dow Corning Antifoam A, but it is especially useful in that it leaves no carryover in food packaging paper or board stock to contaminate or impart an odor to food.

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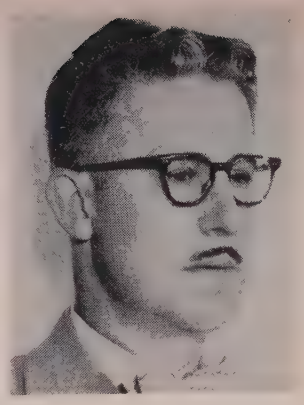
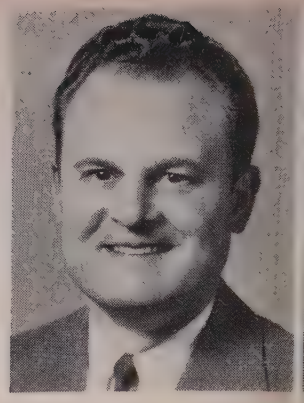


J. W. Couture, The Black-Clawson Co.



K. A. Oberreich, Appleton Woolen Mills

B-C (Shartle & Dilts)

J. W. Couture has been appointed Northeastern Sales Manager of the Black-Clawson Co. In this new capacity Mr. Couture will represent and supervise the sales of the products of all divisions of the company. He has been Chief Engineer of the Dilts Div. at Fulton, N. Y., where he will continue to maintain his headquarters.

Appleton Woolen

Karl A. Oberreich has been appointed Sales Manager of the Appleton Woolen Mills, Appleton, Wis. Mr. Oberreich is a graduate Chemical Engineer having had 22 years' experience in the paper field which comprised technical and research work, purchasing, and selling.

Reichhold

Completing its third major expansion in recent weeks, Reichhold Chemicals, Inc., has opened offices and a laboratory at South San Franscisco, Calif. Reichhold has 2 plants in the United States and is the largest manufacturer of synthetic resins in the world.

American Polymer

American Resinous and Chemicals Co. and the American Polymer Co., Peabody, Mass., have appointed Orville H. Heinicke and F. L. Sutton as Sales Engineers. Mr. Heinicke will represent the company in Ohio and Mr. Sutton in Connecticut and Metropolitan New York.

Pennsalt

Pennsylvania Salt Mfg. Co. has appointed James M. McWhirter, Manager of its Wyandotte, Mich., works, replacing G. A. Nelson who is being assigned to engineering and consultive work. Mr. McWhirter was formerly with the General Chemical Co.

Permutit

The Permutit Co., 330 W. 42nd St., New York 18, N. Y., has issued a new bulletin, No. 2386 Permutit Zeolite Water Softeners.

Blaw-Knox

Blaw-Knox Co., Becket Dept., Pittsburgh 30, Pa., has issued a 12-page illustrated bulletin No. 2397, "Grapples for Pulpwood and Logs."

Lobdell United

George E. Kerr, formerly with Yale & Towne, is now Sales Engineer for Lobdell United Co., Wilmington, Del., subsidiary of United Engineering & Foundry Co., makers of roll, roll calipers, calender stacks, and roll grinders for the paper industry.

Bolton

John W. Bolton Co., Lawrence, Mass., announces that the

winners of its Fifth Bolton Award Contest are: 1st, Hugh A. Black, Mead Corp., Chillicothe, Ohio; 2nd, William H. Chandler, S. D. Warren Co., Cumberland Mills, Me.; and 3rd, C. G. Krancher, Ball Brothers Co., Noblesville, Ind. The subject for this year's composition was "Successful Selection of Supervisory Personnel." The essays will be published in book form.

Antara

Antara Chemicals Div. of the General Dyestuff Corp., 435 Hudson St., New York 14, N. Y., has issued an interesting brochure, "Trouble Shooting with Blancol," describing Blancol conc. powder and fine powder N used in processing paper and pulp. These compounds are used to prevent coagulation of pitch, prevent slime growth, minimize sticking to press, minimize graniting, etc.

Morningstar Nicol

Morningstar Nicol, Inc., has acquired the Jersey City factory and three departments of Innis, Speiden Co. The plants obtained manufacture gum arabic, tragacanth, locust bean, and gum ghatti.

Reliance

Reliance Electric & Engineering Co., 1088 Ivanhoe Rd., Cleveland 10, Ohio, has issued a new bulletin (A-2703) "Motor Drive Is More Than Power."

American Conditioning House

E. Edsall Riley, Jr., has become associated with the American Conditioning House, Inc., Boston textile organization, specializing in laundry and detergent research, located at 11 Melcher St., Boston 10, Mass. The American Conditioning House is a commercial testing and research laboratory.

Ferguson Machine

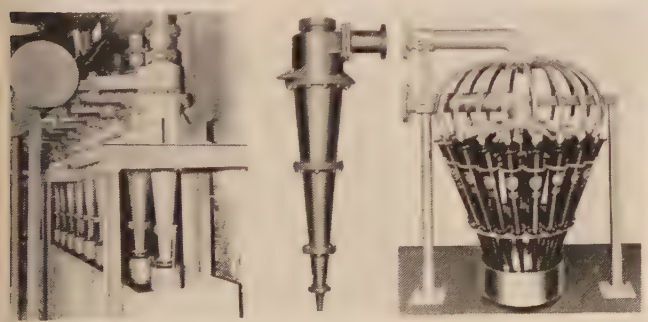
Ferguson Machine & Tool Co., Ferguson, Mo., subsidiary of the Universal Match Co., has issued a 4-page brochure describing its services and a reprint "Designing Cams" by C. N. Neklutin. The reprint is an excellent presentation of the subject.

Diamond Alkali

The Diamond Alkali Co., 300 Union Commerce Bldg., Cleveland 14, Ohio, has issued a new technical manual on "Soda Bleach Solutions."

Bauer

Bauer Bros. Co., 1715 Sheridan Ave., Springfield, Ohio, has issued an interesting bulletin (P-4) on its new Centri-Cleaners, showing the Hammermill and the Howard Smith types.



Left: Battery of Howard Smith type Centri-Cleaners. Center: Howard Smith type Centri-Cleaner. Interchangeable cast-iron sections. Right: Group of Hammermill type Centri-Cleaners connected to inlet and outlet manifolds

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50% water solution of potassium trichlorophenate

Excellent for washups as a slime prevention measure-compatible with scouring agents.

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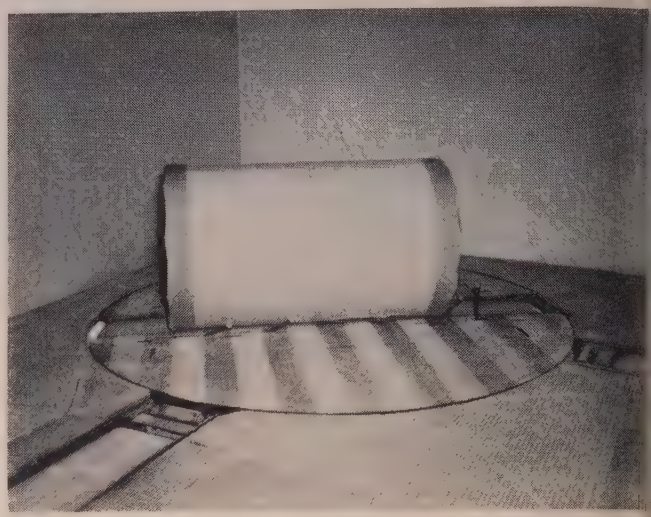
CHICAGO: F. M. deBeers and Associates, 20 N. Wacker

LAMSON

The Lamson Corp., Syracuse, N. Y., now manufactures a turntable to permit rolls of paper to be turned at right angles



Turntable for paper rolls manufactured by the Lawson Corp.

Two-ton rolls can be handled in this way. The device meets obstacles to handling materials set up by building peculiarities

MONSANTO

Oscar P. Cohen has been appointed a group leader in the research and development department of the Merrima Div. of Monsanto Chemical Co., Everett, Wash. Mr. Cohe will specialize on papermaking chemicals.

Monsanto Chemical Co., Springfield, Mass., has issued Production Information Bulletin No. 58 on Lustrex Latices aqueous dispersions of styrene resins.

MORDEN

Blake Honeyman, Vice-President of Morden Machine Co., Portland, Ore., has returned from a trip to Japan where he inspected 26 Morden stock-makers in 12 Japanese mill

LOUIS ALLIS

Louis Allis Co., Milwaukee 7, Wis., has issued a new bulletin (No. 800) covering its line of explosion-proof motors

SANDUSKY FOUNDRY

The Sandusky Foundry & Machine Co., Sandusky, Ohio, has issued a 4-page Specification Bulletin (No. 136) relating to the Chief Sandusky shower.

JACKSON & CHURCH

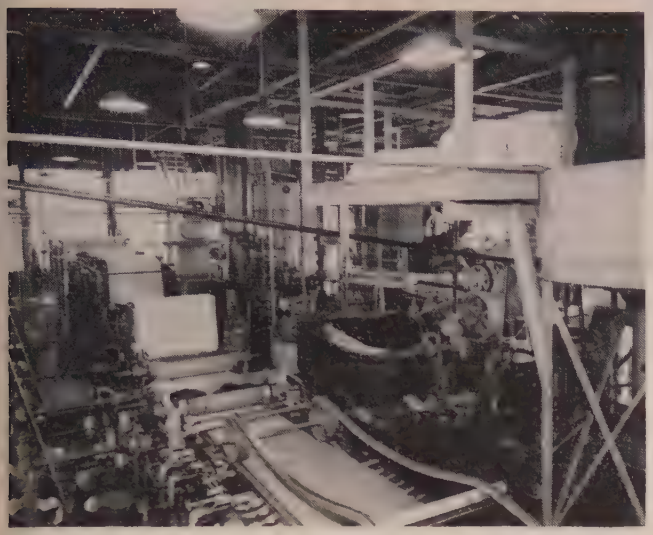
L. F. Reinmann has been appointed Assistant Vice-President of Manufacturing of Jackson and Church Co., Saginaw, Mich.

To assist Herty Foundation Laboratory, Savannah, Ga., and University of Florida, Gainesville, Fla., in carrying out an expanded experimental program with paper pulp, Jackson & Church Co. has furnished some of its newest processing equipment. The laboratories recently have stepped up experimentation with all types of wood pulps and fibers in an attempt to find acceptable and profitable uses in the paper industry. The results have been hailed as major contributions toward finding new sources of wood pulp to meet the ever-growing paper needs of the world.

A Zenith Pulp Press, used for obtaining pulp consistencies up to 50%, and an 015 refiner-shredder-mixer, recently designed for introducing bleach to high consistency pulp, were provided Herty Foundation. An 015 machine has been installed at the University of Florida and a press similar to

the one at Herty also is to be made available in the near future.

Herty is set up to carry out pilot plant experiments for firms in the cellulose field. It is using the 015 for decorticat-



Overall view of the paper machine in the pilot plant of the Herty Foundation Laboratory

ing bagasse, flax, and similar agricultural fibers as well as a bleach mixer for high density problems and studies on refining of Southern unbleached pulps. Zenith presses are used for dewatering pulp and as washers. Herty has other equipment designed to handle paper pulp problems on a pilot plant scale under direction of Reavis C. Sproull, technical director, and Walter L. Hendrix, operational director.

Jackson & Church engineers, in conjunction with one of the leading paper firms and Herty personnel, recently helped carry out complete tests on high density bleaching, the results of which were termed "highly satisfactory."

The University of Florida is currently using the 015 as a refiner, particularly on semichemical processes. It is also to be used in hot refining of kraft pulps and as a mixer for bleaching at high density. Its adaptability as a shredder for presoftened chips, prior to continuous cooking, is to be investigated. The Zenith press will be used as part of the 3 to 6 ton per day pilot plant to be installed in the laboratory for further exploration of continuous pulping, according to Dr. Wilson F. Brown, in charge of pulp and paper research, and Dr. William J. Nolan. The laboratory, a part of the Chemical Engineering Section of the Engineering and Industrial Experiment Station, is now designed primarily for carrying on fundamental and applied research in pulp and paper.

John Waldron's 125th

Probably no manufacturing enterprise in the world today has had so active a part in the development of a single phase of one large industry as has the John Waldron Corp., New Brunswick, N. J., who has concentrated in the paper converting field, and who is this year celebrating the 125th Anniversary of its founding; starting in the year 1827 in New Brunswick, then a small town and initiating its industrial career as a leather currier, the impetus to branch out into other fields came with the installation of one of the early wood and metal turning lathes for making shoe lasts in the interest of promoting its leather tanning business.

In 1840 Pratt and Hardenberg, later succeeded by Janeway and Carpenter in New Brunswick and M. A. Howell and Co. were making wallpaper printed by hand with wood blocks. Howell & Co. imported a single color printing machine from England, which could print as many as 200 rolls per day by the surface printing method and to compete with this "High Speed," Waldron was instructed to build a machine grounder

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Complete range of butadiene-styrene copolymers from 100% butadiene to 100% styrene . . . and metal ion resistant types . . . recommended for paper coatings, water paints, textile finishing, paper saturation, adhesive bases . . . Bulletin P-24, P-29.

ACRYLIC EMULSIONS

Series of acrylic copolymer emulsions which deposit clear, tough flexible films with outstanding ageing characteristics . . . freedom from plasticizer insures constant film properties with no embrittlement, spewing or migration . . . products used for leather finishing, paper coatings and saturation, textile finishing, adhesive bases, protective coatings . . . Bulletins P-4, P-23, P-25, P-28.

ACRYLIC SOLUTIONS

Acrylic polymers and copolymers in various solvents . . . deposit clear, water-white films ranging from tough, hard to soft and rubber-like . . . resistant to water, alkalis, acids, mineral oils . . . low pigment reactivity . . . excellent base for compounding adhesives, protective and functional coatings . . . compatible with wide range of lacquer and paint resins . . . Bulletin P-26.

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Vinyl chloride copolymers yielding tough, elastic films without plasticizers . . . excellent oil and chemical resistance . . . low moisture vapor permeability . . . recommended for leather finishing, paper coating, adhesive bases . . . Bulletins P-32, P-33.

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Stable aqueous solutions used as thickening agents for natural and synthetic latices . . . particularly valuable for adjusting viscosities of water based paints . . . Bulletin P-27.

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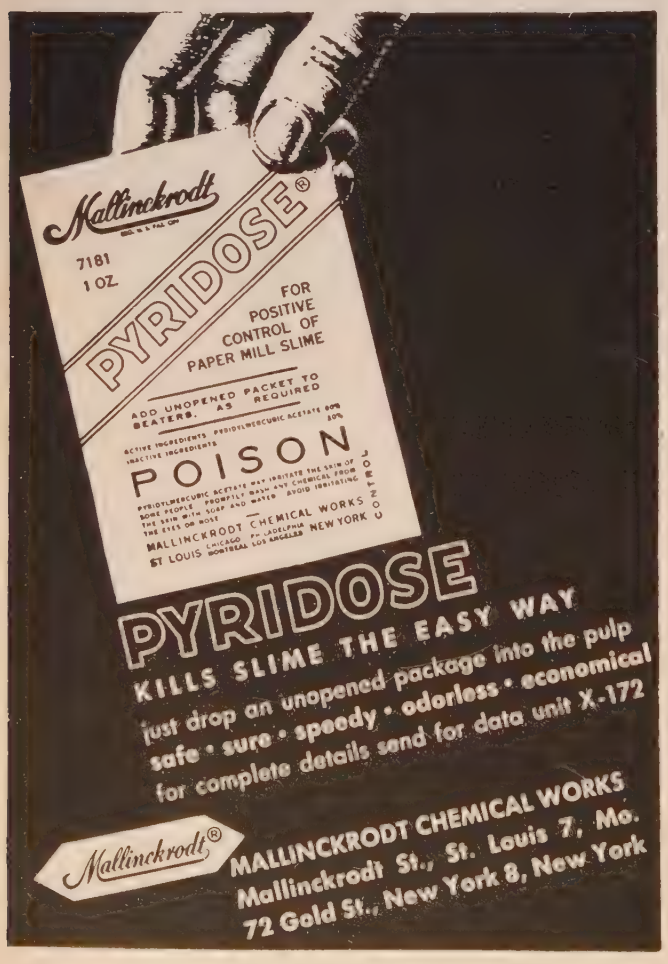
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or coater to improve the surface of the paper to give it better receptive quality for the color; for this purpose the power-driven oscillating brush coater was developed and at the same period in 1848 a multiple color printing machine was designed and built for Pratt and Hardenberg, as well as a machine for hanging up and carrying off paper hangings as the patent describes it. This was in the 83rd year of the independence of the United States of America and since 1827 the company has been continuously in business under the name of Waldron. The design of the printing machine was then improved and in 1854 higher speeds of printing were attained and the number of colors was increased to eight and in 1855 to the standard twelve color machines now universally used. With this increase in number of colors and rates of production, the demand for better coatings and quicker drying was constantly increasing, in order to keep up with the production of the many printing machines, which were being built by Waldron.

By this time Waldron has built about 15, 8 and 12 color printers and it also built power-driven oscillating brush coaters and festoon racks for drying the ground-coated and printed paper and was exporting printers and grounders to England, who then re-exported the products of these machines to the United States.

Papermaking at this stage in history was an art and not a science as it is today and the preparation of the surface to provide good printing ink reception, except by hand, was practically unknown in the fine paper field, but in 1852 at the solicitation of Louis DeJonge and Co. of Staten Island Waldron agreed to build and deliver the first paper coating machine for fine printing and writing papers and guaranteed its satisfactory operation.

Unusual though it may seem two of these early machines built as long ago as 1872 are still in operation. One of these is in a New England paper converting mill and its mechanism is carried by a wooden frame, this machine feeds into the old style hanging machine and it is still producing good coated paper on special runs; at the same company in a new building is housed one of Waldron's ultra modern Microjets capable of operating up to 1000 f.p.m. with automatic splicing and both unwind and reroll ends of the machine.

Early in the 1840's further iron working tools were acquired and also a steam engine installed to drive them. This was about the time when the first telegraph line in the United States was established between Baltimore, and Washington and the first American built all-metal steam-driven boat, which was launched in Pittsburgh, Pa., and not long after the first steam-driven train in the United States made the journey from Albany to Schenectady.

At this period the wallpaper industry was in its early stages of production in this country and it really became the forerunner of machine-operated paper coating.

Waldron has pioneered in the development of the reverse roll coater, the offset gravure printer type of coater, laminators for foil paper, and high-speed printing machines for plastic film as well as casting, printing, coating, and saturating equipment for many different types of materials in web form.

Today Waldron is the largest producer of paper, plastic and textile converting machinery in the world, and its name is synonymous with paper converting developments. It also leads in the field of special printing equipment, and four new developments have entered the field this year as the result of its continuous progress in research. These are a four-color printer for cylindrical steel or fiber barrels, a high-speed automatic web-fed multicolor offset printing press delivering $17\frac{1}{2}$ by $22\frac{1}{2}$ sheets at 14,000 impressions per hour, a new paper plastic and textile multicolor printer with running for pattern and a low-priced coater for performing several different types of coating operations on the one machine by simple change-overs.

During its 125 years of experience, the John Waldron Corp

has gathered a tremendous body of knowledge relating to paper treatment and conversion. Engineering departments of various concerns frequently call on Waldron's president, B. R. Newcomb, for information on converting operations which is unavailable in the records of other institutions. This carefully kept historical data often save expenditures for duplicating research which has already been performed.

HUBINGER

A review of the company's recent expansion program and a comprehensive outline of sales objectives and marketing problems featured the national sales meeting of The Hubinger Co., held recently at Keohuk.

Hubinger Co. executives and sales personnel at the company's recent national sales meeting. Front row: R. L. Fisher, President: Howard Jackson, Secretary; E. J. Carlson, D. L. Edwards, Frank Craven, Donald Banghart, Howard Richmond, and A. M. Robinson, Vice-President in charge of sales. Second row: H. E. Myers, Harry Wischmann, John Mulberry, George Hines, W. C. Fletcher, Sales Manager of packaged goods division; Henry Dusick, John Wallenbrock, Herbert Lawrence. Third row: Frank West, D. L. Tiger, W. B. Strickland, L. G. Drusendahl, Howard Bentz, J. P. Fanning, Everett Humble, Howard Peper. Back row: R. L. Kreuger, Assistant to the President; William Nye, J. T. Flahiff, L. C. Watson, Vice-President in charge of production; G. R. Underwood, Sales Manager of bulk products division; L. F. Adelberg, and W. M. Wolf

President Robert L. Fisher and Roy L. Krueger, the company's treasurer, traced the company's development and modernization program. Improvements to plant since the war, they said, have added materially to plant capacity, making it one of the most efficient units for its size in the corn refining industry.

Arthur M. Robinson, vice-president in charge of sales, gave the broad picture on current sales and objectives. Panel discussions for bulk products and consumer packaged goods were led by W. C. Fletcher, sale manager for the packaged goods division, and G. R. Underwood, sales manager for the bulk products division. Some 30 salesmen and company executives participated in the two-day meeting.

RESEARCH CORP.

Initial operation of their first new wet-bottom type Cottrell electrostatic precipitator has been reported by Riegel Paper Corp. The new precipitator was supplied by Research Corp. for Riegel's new mill in Acme, N. C., which is reputed to be the world's most completely instrumented mill.

The wet-bottom design is a new development in precipitators for paper mill applications. Utilizing a circulating black liquor flow, instead of dry dust hoppers and conveyors, a wet-bottom dust handling system allows rapid re-use of the collected dust. It also eliminates auxiliary equipment

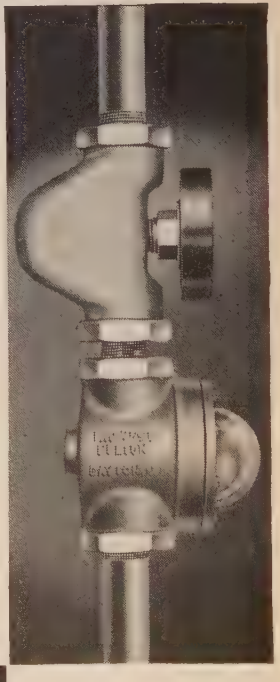
Test and Record Stiffness of Most Papers and Paper Boards in 21 Seconds

That's all it takes to measure paper up to 0.25" thick with the motorized Gurley Stiffness Tester. It easily handles tissue, newsprint, book, bonds, ledgers, industrial papers, cardboard and carton stocks, and permits a wide range of readings from 1 to 3,358,720.

More than 300 of these aids to faster, more efficient measurement are used in leading mills and laboratories.

Write for Bulletin No. 1400. It describes the motor-driven and hand-operated models as well as other Gurley paper-testing instruments. W. & L. E. Gurley, Station Plaza & Fulton Street, Troy, New York.

A Sight-Flow Indicator *that Really Works*



The Midwest Ball Sight-Flow is the one and only dryer condensate indicator with a "Window" that really stays clear—is constantly being scoured and kept clean by the violence of the liquid as it passes. The thousands already in use on both wet and dry end dryers prove that statement.

Flow visible from front and both sides. Installation in discharge line either vertical or horizontal.

Equip your dryers, particularly the wet end section, and keep tab on condensate flow. Add Midwest non-corrosive temperature indicator to help you spot subnormal temperatures.

Sight-Flow fitting all bronze and non-corroding. Glass ball of heavy Pyrex glass and replaceable. Assembly effective for steam pressures up to 75#. Standard pipe sizes ½", ¾", 1", 1¼", 1½". Temperature indicator also non-corroding.

Order several now and you'll order more later. They do the work or we take them back.

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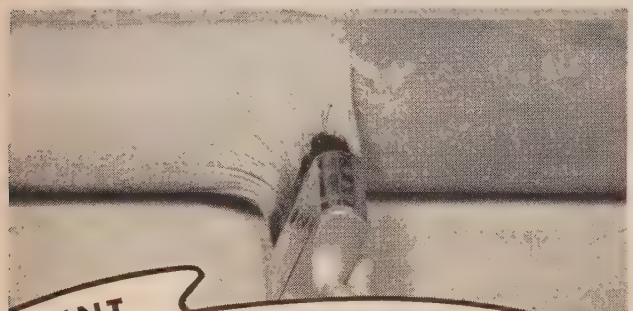

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- inhibits rust formation

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Akron 9, Ohio

formerly used to prevent moisture condensation in the hopper and in the handling of the collected material between the hoppers and dissolving tank. The wet-bottom precipitator (patents pending) was developed by Research Corp., Bound Brook, N. J., manufacturer and designer of many "first" Cottrell precipitator applications in a wide range of industrial jobs.

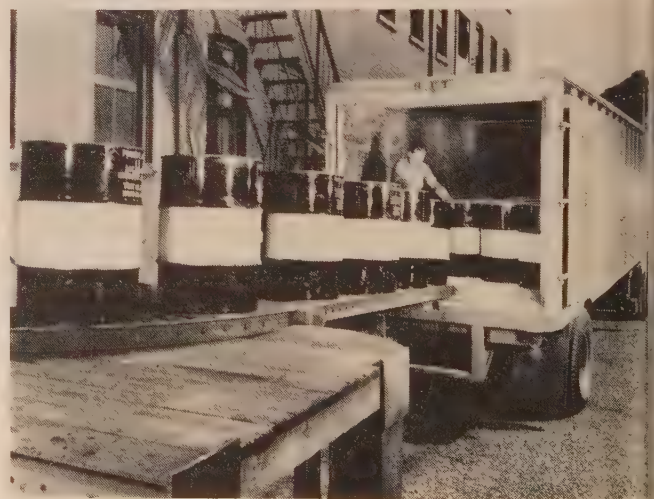
At the Riegel Paper Corp. plant in Acme the new precipitator removes sodium sulphate from gases coming from a spray-type recovery furnace handling 750,000 lb. of black liquor solids per day. On installation, the precipitator was designed to clean 112,000 c.f.m. of gas at 280°F. at a rated efficiency of 90%, but operation reports show that actual recovery efficiency is 95%.

Initial operation of their first new wet-bottom type electrostatic precipitator has been reported by Mead Corp. The wet-bottom design is a new development in precipitators for paper mill applications. Utilizing a circulating black liquor flow, instead of dry dust hoppers and conveyors, a wet-bottom system enables rapid re-use of the collected dust. The new precipitator (patents pending) was developed by Research Corp., Bound Brook, N. J., initiator of many "first" Cottrell precipitator applications in a wide range of industrial activities.

The new wet-bottom precipitator at the Mead Corp., Chillicothe, Ohio, plant removes sodium carbonate from gases leaving a spray-type recovery furnace used in processing black liquor in soda pulp making. The furnace was designed to process approximately 525,000 lb. of black liquor solids daily. At rated capacity the precipitator will clean 61,250 cu. ft. of gas at about 300°F. with 95% rated efficiency. Under present mill operating conditions, 350,000 lb. of black liquor solids are being processed daily with a gas volume through the precipitator of about 44,000 c.f.m. at 270°F. Under these conditions, an efficiency in excess of 97% is being realized.

NOPCO

Off the automatic conveyor and into the transportation company's giant truck rolls the first load of Nopco Chemical Co.'s newly adopted "dressed up" drums. Swing-over to



First load of Nopco black and orange drums leaving shipping platform

the more distinctive, more colorful package by the Harrison, N. J., concern promises a twofold advantage to Nopco customers. The sparkling black drum with its brilliant golden-yellow center band will make quick identification of Nopco products possible in customers' plants and warehouses. Additional stenciling of all-important shipping marks on the bung sides of the drums, as well as on the heads, is expected to prove a distinct advantage particularly to those customers

who are warehousing in palletized units. Nopco officials, in announcing adoption of the new drum design, state that extensive use of the drums has already begun and that all existing drums will soon be replaced with them.

THEW SHOVEL

A special front end attachment perfected for unloading pulpwood rack cars, called the Lorain Rake, has just been announced by The Thew Shovel Co., Lorain, Ohio.

The Lorain Rake announced by The Thew Shovel Co.

The new Lorain Rake is now available for use on $^3/_4$-yd. Lorain TL-25 models on crawlers or rubber-tire carriers. Equipped with a gooseneck boom its maximum reach of 31 ft. can unload a car in from 10 to 12 minutes—or as fast as the flume or conveyor can take the logs. The Rake has a horizontal pulling action toward the machine which provides positive and precise control for spotting the 5 by 3-ft. board. Operated on the principle of a parallelogram, the board maintains a semivertical position at all angles of the boom and stick. Complete visibility allows placing of the board exactly where it is needed and it may be moved in any manner toward or away from the machine through a simultaneous drag and swing action. This enables the car to be quickly and completely cleaned with a minimum of manual clean-up. This precise control minimizes car damage and pulpwood spillage with its attendant added labor cost.

D. J. MURRAY

Shown sealing the cornerstone of the new two-story brick addition, 126 by 115 ft., to their manufacturing plant is A. W. Plier, Executive Vice-President, General Manager of

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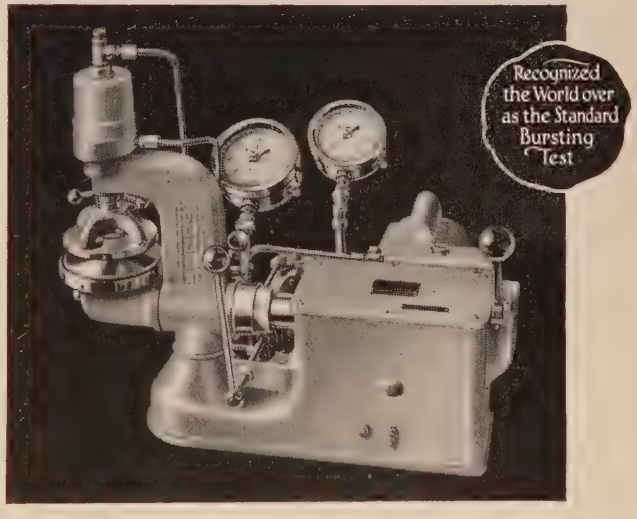

Especially designed for testing corrugated container board. Larger clamping area and reproducible clamping at any desired pressure eliminates crushing and slippage of the sample.

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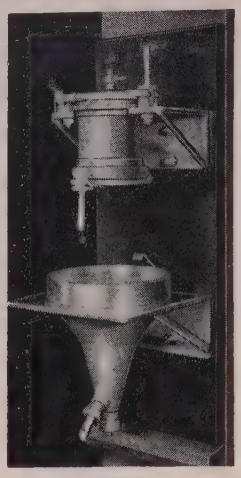
for accurate beating control



Tappi Standard T 227 M-50

The Spotlight is on

TESTING MACHINES, INC.

manufacturers and distributors of the finest test equipment for the paper, textiles, plastics, metals industries and laboratories.

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the D. J. Murray Manufacturing Co., Wausau, Wis. The addition is concrete and steel faced with brick exterior walls to match the remainder of the plant. Fred C. Boyce, company president (left), gave the dedication address during the program for dedicating the new addition which was held early in September in conjunction with the company's 70th anniversary as a manufacturer. About 750 persons attended the dedication, including officers, directors, stockholders, employees of the company, and civic representatives. The picture was taken on the Fulton St. side of the building that is on the corner of Fulton & Third Sts. D. J. Murray Manufacturing Co. manufactures a specialized line of paper mill equipment, unit heaters and blast coils, power dam gates and gate hoists, grease traps, and a varied line of special machinery.

Pandia's 20th Anniversary

Pandia, Inc. was incorporated on Sept. 20, 1932, under the name of Paper and Industrial Appliances, Inc. The company founder was R. D. Kehoe who was President and directed the company's activities until his death in December, 1949. The company name was changed to Pandia, Inc., on Jan. 1, 1952.

Pandia, Inc.'s entire history has been in the role of a supplier of equipment to the pulp and paper industry. The company has sold and engineered a wide variety of equipment for the pulp and paper industry during its lifetime and has steadily progressed with the developments in the industry.

Two important pieces of equipment now available to the pulp and paper industry are the direct result of the pioneering and development work carried on by Pandia, Inc.

The Brammer recording consistency control which is considered one of the best on the market in its field, has been steadily developed and improved over the years.

The Chemi-pulper continuous digester is a development of Pandia, Inc. Early uses of the Chemi-pulper were in the insulating board and the roofing and flooring felt industries. The Chemi-pulper advanced in the corrugating board field with the development of semichemical pulps for that industry. Pandia, Inc. has developed and is improving the design of the Chemi-pulper to the point where it is possible to custom build a continuous digester to meet a user's specific requirements.

Pandia, Inc.'s activities are under the direction of the following: A. Whittemore, President, L. G. Durant, Vice-President, and R. G. Goodwin, Director, Sales Engineering.

Pandia, Inc. has been successful and has kept pace with progress during its first 20 years, and now plans to go forward at a more rapid pace primarily in the development of more and better continuous pulp mill operations.

Rice Barton

Rice Barton Corp. has started construction of new facilities and additional assembly space which will add 25,000 sq. ft. to their present manufacturing area. The photograph below showing Charles S. Barton, President, Angus J. Gardner, Vice-President in Charge of Sales, Everett W. Clem, Vice-President in Charge of Engineering, and Howard I. Eddy, Supervisor of Production, shows a portion of this expansion. The principal new components of this area will be:

1. A one-story steel and glass building to be used for manufacturing facilities and small assemblies.
2. A new manufacturing building approximately 40 ft. wide by 210 ft. long for the manufacture of paper machine driers with facilities capable of handling rolls 72 in. in diameter by 300 in. in length.
3. A roofed area between its existing pattern storage building and its existing lathe building. This area, approximately 100 by 30 ft., is adjacent to the foundry and will be used for additional chipping and cleaning space.

Additional facilities will make room for new dynamic roll balancing machinery which is the largest of its kind ever to be built. It is capable of the utmost accuracy in dynamic balancing of rolls up to a surface speed of 7000 f.p.m. and is designed to handle rolls 300 in. long.

Other equipment which will be installed includes a 6 in. bar horizontal boring machine, several smaller horizontal boring machines, a large planer, a large surface grinder, and other smaller machine tools.

Approximately a hundred or more machinists are being recruited in the Worcester area which is noted for its production of machine tools to man the expanded production facilities.

Among the orders which Rice Barton has on their books is a pulp machine for Ketchikan Pulp Co. of Ketchikan, Alaska—the first installation of the pulp and paper industry in Alaska. It is also building a pulp drying machine for Rayonier, Inc., for their new mill in Doctortown, Ga.

View of the new construction for the Rice Barton Corp.

TAPPI DIVISIONS AND COMMITTEES

Reports of Activities

Testing Division

The following reports by chairmen or secretaries of the committees of the Testing Division of the Technical Association refer to the subjects considered at the TAPPI Testing Conference held at Syracuse, N. Y., on Sept. 10–12, 1952.

Pulp Testing Committee

The sessions of the Pulp Testing Committee were held in Marshall Hall, New York State College of Forestry, at Syracuse University. Thirty-four were in attendance. The discussions were divided into three general groups for consideration.

1. Review of work completed and in progress. This included a discussion of the Authorized Committee Projects as follows:

No. 132, Revision of T 200 m-45—Laboratory Processing of Pulp (Beater Method). The following points were discussed and changes will be submitted for committee review.

(a) Grinding procedure is to be expanded to provide information on the use of finer grits for smooth polishing and drilling.
(b) Projection of the first bedplate bar was discussed. It was agreed that this should be specified as at least $^1/_8$ in.
(c) Bedplate bar dimensions are to be specified to a closer tolerance. The supplier is to be consulted.
(d) Calibration procedures were discussed. It was recommended that notes on methods used be incorporated in the method.
(e) It was recommended that wording be changed to specify roll speed "under load."

No. 140, Shives in Pulp. The discussion of this proposed method was held over to discussion under Dirt in Pulp.

No. 333, Revision of T 205 m-50—Forming Handsheets for Physical Testing of Pulp. The insertion of alternate procedures for vacuum couching and automatic pressing was presented for discussion.

The wording on vacuum couching was revised and recommendation made that additional information be provided to standardize the equipment arrangement and vacuum. It was pointed out that a round blotter is required when using this procedure.

The recommended change on automatic pressing was approved.

No. 334, Revision of T 226 sm-46—Specific External Surface Pulp. This revision has been approved by the Standards Committee. The changes involved were discussed.

No. 335, Fiber Classification. The proposed basis for this method was presented by Dr. Clark. A draft is in preparation and will be circulated for committee approval

The method, Zero Span Tensile Test for Pulp, was presented in its rough draft. This draft will be brought up to date with regard to equipment specifications and submitted to the committee for approval.

2. The second session was devoted to discussion on revision of T 213 m-39—Dirt in Pulp.

A review of the work on this method was presented by the chairman. Following this, the basic requirements chosen for this method were discussed, point by point. These requirements were embodied in the report made by the Subcommittee on Dirt at the National Meeting. After extensive exploration of all points, it was agreed to accept these requirements on the following basis:

The developed procedure, modified as recommended, will be provided as a basis for tests to be made in several laboratories. The tests will be set up in such a way as to provide data for statistical analysis. After such analysis, it will be possible to evaluate the majority of points in question, and proceed with the final draft of the revised method.

In connection with this method, there was considerable discussion on Shives in Pulp. It was agreed that a count of shives should be included in the Dirt Method. A definition of the term "shive" was developed as follows:

Shive: Incompletely defibered woody material that has a contrasting color with the pulp sheet and is 0.04 sq. mm. in area or greater.

3. The third session was conducted immediately and included discussion of new work planned by the committee.

The first of these proposed projects was Consistency of Pulp. After discussion it was agreed:

(a) There is some question whether such a method belongs in the TAPPI methods.
(b) Techniques of sampling and handling are the real problems involved and, for the establishment of a referee method, are extremely difficult to define.
(c) The need for such a method is in connection with the delivery of pulps in slush form.
(d) Further discussions will be conducted by the Pacific Section Subcommittee.

The second proposed project was a revision of T 210 m-45—Weighing, Sampling, and Testing Wood Pulp for Moisture. After discussion it was agreed:

(a) Rewrite paragraph 4 (under Apparatus) to specify the equipment requirements more closely and change the temperature to 105°C. ± 3°.
(b) Prepare a proposal for interested groups regarding an independent investigation and statistical analysis of sampling procedures.

A proposed suggested method for the Fineness of Pulp was discussed. It was agreed to prepare a draft of such a method for submission to the committee.

The establishment of a standard pulp service by TAPPI was discussed in a preliminary way. The purpose of such a service would be to make available a pulp for standardization of pulp evaluation equipment in the individual laboratories.

The sessions of the committee were well attended by a representative group from both pulp users and producers. Almost all of those present participated in the discussions.

R. I. Theime, *Chairman*
Pulp Testing Committee
Scott Paper Co., Everett, Wash.

Nonfibrous Materials Testing Committee

The following is a report of the present status of the projects under the jurisdiction of the Nonfibrous Materials Testing Committee and covers the work done at the Testing Conference in Syracuse.

Enzyme Testing: The Enzyme Testing Subcommittee under the chairmanship of Dr. Frieden has just about completed its work on an enzyme testing method. The procedures need to be written up in TAPPI form and a decision reached as to which one of the various procedures for testing

for enzyme activity should be used. Both photometric and viscosity methods are being considered and the present plan is to select one photometric and one viscosity method and write each of these up as a separate procedure. The result can then be reported as enzyme activity by photometric or viscosity method, leaving the choice of which method to use up to the analyst. It is expected that the methods will be written up in their final form in the very near future so that they can be submitted to the Standards Committee.

Rosin and Rosin Size Testing: A report on the Rosin and Rosin Size methods was given at the time of the Syracuse meeting by Mr. Cameron, who is chairman of the committee for revising these two methods. The method for rosin analysis has been completely rewritten and needs only to be edited before submission to the Standards Committee.

The method for rosin size analysis has been rewritten and several changes made to correct for objections raised by members of TAPPI. The principal change in the method was one providing for the testing of free rosin. The method as now written for free rosin permits the use of either the titration or the extraction method of analysis but states that the method used must be reported by the analyst. Conversion factors are given for reporting free rosin by extraction, using the titration method for analysis.

Mr. Cameron's committee is undertaking the preparation of a method for testing rosin-wax size combinations. This method is intended to be used as a routine control test.

Clay Testing: The Clay Subcommittee under the chairmanship of Dr. Albert held a number of meetings at the time of the Syracuse Conference to discuss the various procedures to be included in a method for analysis of clay. The various procedures for which write-ups have already been obtained include sampling, moisture content, pH, brightness, viscosity of coating clays, grit, disintegration of coating clays and particle size distribution. Other procedures including alkalinity, abrasiveness, and hardness are being considered for the routine control tests. Considerable discussion revolved around the best method for testing for particle size distribution of clays. There are several methods being used at present and the results obtained by the different methods do not always agree. Some work has been done cooperatively by members of the committee to reconcile the results obtained by these different methods for measuring particle size, but it was decided that further work needed to be done before a satisfactory conclusion could be reached. The Clay Committee is now outlining the work which is indicated for particle size measurement, and it is planned to have this work done in the very near future so that a completely satisfactory method for measuring particle size can be prepared soon. In the meantime, it was decided to proceed with the preparation of the other methods for analyzing clay, submitting these to the Standards Committee as soon as possible.

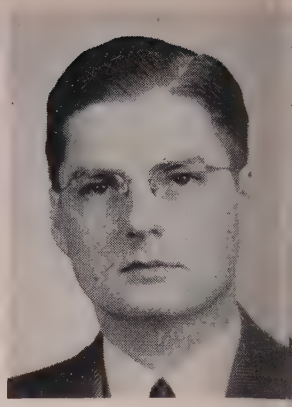
Titanium Testing: At the time of the meeting in Syracuse it was agreed that a Titanium Testing Subcommittee should be organized to prepare methods for the analysis of titanium pigments. Mr. Willets is chairman of this committee and will undertake to organize a committee membership in the near future.

Starch Testing: The work of the Starch Testing Subcommittee is complete and the method has been written up in its final form. It is now being typed for the final time before submission to the Standards Committee.

Calcium Sulphate Testing: A rewrite of TAPPI Method T 615 to include calcium sulphate only has been prepared by John Lewis. This method needs to be edited and then it is ready for submission to the Standards Committee.

Casein Testing: The Casein Subcommittee under Mr. Laird is proceeding with the rewriting of the present casein method to include the latest procedure used for analyzing this raw material. Members of the Casein Committee have been requested to submit their opinions of the present method, and this work is progressing slowly.

R. I. Thieme, Scott Paper Co., Chairman, Pulp Testing Committee



J. P. Casey, A. E. Staley Mfg. Co., Chairman, Nonfibrous Raw Material Testing Committee

Animal Glue Testing: It has been agreed to set up a committee for preparing a method for the analysis of animal glue. This subcommittee has not been organized as yet and any members of TAPPI interested in serving on this committee will be welcome.

JAMES P. CASEY, *Chairman*
Nonfibrous Materials Testing Committee
A. E. Staley Mfg. Co., Decatur, Ill.

Microscopy Committee

Two sessions of the TAPPI Microscopy Committee were held during the recent Testing Conference at Syracuse. The first session opened with a brief discussion of the projects listed for the Microscopy Committee in the *TAPPI Yearbook* and new projects now under way. It was reported that the preliminary work on the proposed Microscopy Monograph was being completed under the direction of F. L. Simons and it was hoped that writing of the various sections would be started soon. There was a brief discussion of the need for a detailed study of weight factors and also the need for more work on such fibers as bleached groundwood and semichemical pulp. It was reported that a method for "Microscopical Identification of Papers Fillers" was being prepared by H. O. Koch and a "Method for Identification of Synthetic Fibers" by F. L. Simons.

There was a brief discussion of the various methods now under the jurisdiction of the Microscopy Committee. It was pointed out that the primary requirement for revision of T 9 and T 10 was new photomicrographs. Possible sources for these photomicrographs were discussed and the new edition of the handbook of wood fibers just issued by the New York State College of Forestry was examined.

The procedure for "Identification of Specks and Spots in Paper" was next reviewed and each member of the committee was asked to send in his procedure for identification. The group then used the remainder of the two sessions in making the final corrections on the proposed revision of T 401 m-52 on Fiber Composition of Paper. This method will soon be ready to be presented to the Standards Committee.

C. E. BRANDON, *Chairman*
Microscopy Committee
Howard Paper Mills, Dayton, Ohio

Chemical Methods

The Standards under the jurisdiction of the Chemical Methods Committee include methods for wood, pulp, and paper. Two wood methods were discussed at the Syracuse meeting.

I. Wood Methods

The Methoxyl Groups in Wood, T 2 m-43. E. P. Samsel of Dow Chemical Co. was appointed by the committee to revise the method. It is probable that only minor changes are necessary. There is a great deal of background experience from the determination of alkoxyl groups in cellulose derivatives which can be drawn upon. Mr. Samsel was requested to consult with workers at Hercules Powder Co. to incorporate all recent advances into the revision of T 2 m-43.

Moisture in Wood Chips and Sawdust by the Toluene Method, T 3 m-44. Dr. Broughton presented a toluene method for the determination of the moisture content of certain papers (discussed below). Because of his experience with the toluene technique he was appointed to check T 3 m-44 for the committee and revise it along the lines of the newly proposed Toluene Method for Paper.

II. Pulp Methods

There are a large number of pulp standards under the jurisdiction of the Chemical Methods Committee.

Ash in Pulp, T 211 m-44. Although the ash method appeared to be very simple and straightforward there are many points of technique which came out in the discussion of the method. The loss of the easily sublimed constituents of the ash, the exact furnace temperature to use, and the length of the heating period were all discussed by the various committee members and visitors. The most generally accepted temperatures proved to be 750–800° C. and the time of heating should be set at either 2 or 3 hr. It was the consensus of the group present that platinum dishes should be specified although some workers use porcelain ware. An electric oven will also be specified in the new revision. The size of the sample to be used is a point of very great significance especially when highly purified pulps are being analyzed. The wording of the new standard will call for 5–20 grams of pulp or a sufficient sample to give at least 10 mg. of ash. Mr. Ramsey of the Brown Co. was appointed to bring together the points of the discussion and submit a revision of T 211 m-44 by November 1.

Permanaganate Number of Pulp, T 214-50. This standard will be discussed again at the February, 1953, meeting. It was decided that a moisture-free sample should be used for the test, with a note indicating that both water-wet and air-dry samples will yield results which are different from the moisture-free sample. The advisability of increasing the excess of 0.1 *N* permanganate to broaden the scope of the method was discussed.

The Crown Zellerbach Corp. sent in a concrete suggestion on this point [see also Bourgon, Roger, *Pulp Paper Mag. Canada* 47, No. 3: 125–128 (1946)]. It was suggested that the permanganate number might be most valuable as a test in itself rather than merely a test for the bleachability of pulp. Some members of the Pulp Testing Committee were also present during the discussion and agreed that the table, showing the conversion of permanganate number to chlorine number should be greatly modified or deleted. These conversion values are not nearly so accurate or so fixed as is implied in the present write-up. Mr. Thieme, Chairman of the Pulp Testing Committee, agreed to cooperate with the Chemical Methods Committee in revising the paragraph entitled "Additional Information" in which the conversion of the permanganate number into terms useful for pulp bleaching is to be discussed. A revision of the Standard at the February, 1953, meeting is planned.

Lignin in Wood Pulp, T 222 m-50. There was a rather lengthy discussion on the determination of lignin in wood pulp. The method is now in the process of being revised and will shortly be out of the hands of the committee. However several shortcomings of the method were discussed at the Syracuse meeting. At the suggestion of Dr. Browning a note will be added indicating that the method does not determine the total lignin but only that which is insoluble in the sulphuric acid treatment used. The difficulty of filtering is a serious problem in many cases. Two helpful suggestions were received on the filtering of finely dispersed lignin suspensions. Both involved the transfer of the bulk of the clear liquid through the filter before transferring the solid, and were accomplished by slightly different techniques. The third one involved the siphoning over of the supernatant liquid prior to transferring the lignin residue. A suggestion on improved filtering will be incorporated in the new revision.

The spectrophotometric method for lignin based upon absorption in the ultraviolet was also discussed. A somewhat empirical method for highly refined pulp was also advanced which is based upon the color produced by treatment with sulphuric acid under carefully controlled conditions. This latter method was recommended only for pulps having a very low lignin content; so small that a precise answer by the gravimetric method is difficult or impossible.

Alkali Solubility of Pulp T 212 m-44: One Per Cent Caustic Solubility of Pulp, T 212 m-44, Alpha, Beta, Gamma-Cellulose in Pulp, T 203 m-44 were discussed at some length. Other organizations and companies recommend still other alkali solubility tests. There is a 10% potassium hydroxide (7.14% sodium hydroxide) test sponsored by the American Chemical Society and still other strengths of alkali are used. It was suggested that hot and cold extraction of pulp with alkali could furnish valuable information but that the present methods were not satisfactory. Dr. Martin pointed out that in the present hot method, T 212 m-44, One Per Cent Caustic Solubility of Pulp, a considerable portion of the extractable material was actually formed during the extraction procedure by the attack of oxygen on the cellulose in the presence of the alkali. A vacuum technique to avoid exposure to air was suggested and will be incorporated in a revision of T 212.

In connection with the cold extraction the need for a method to determine the yield of product in the viscose industry was emphasized. The present alpha-cellulose method does not appear to be satisfactory since it measures solubility at 8.3% sodium hydroxide after a preliminary treatment with 17.5% sodium hydroxide instead of the direct solubility in the mercerizing strength alkali. The problem of alpha-cellulose, however, must be solved in conjunction with ACS and ASTM and it does not seem likely that the method finally approved will depart materially from the classical method for alpha-cellulose.

Dr. Martin suggested that consideration be given to the colorimetric method based upon the reagent anthrone. An aliquot portion of the filtrate from a cold alkali treatment (without washing) could be analyzed with anthrone to give a value for the solubility in 17.5% sodium hydroxide.

There was also a long discussion on the relative merits of the gravimetric and the volumetric method for the determination of beta- and gamma-cellulose. The present method T 203 does not contain directions for determining beta- and gamma-cellulose. The new revision will contain such procedures which will be worked out in cooperation with ACS and ASTM. The chairman appointed J. W. Jacokes of the American Enka Corp. to the alpha-cellulose subcommittee. He was asked to make a rough draft of a beta- and gamma-procedure to be added to the T 203 standards.

Disperse Viscosity, T 206 and T 230. A very excellent summary of the work being carried on by the viscosity subcommittee was given by its chairman, Dr. Martin. He described the progress made by Dr. Browning who is working on a research grant sponsored by our committee. Dr. Martin, who also heads the viscosity subcommittee for the American Chemical Society and ASTM outlined the topics to be investigated during the coming year under an extension of the TAPPI grant. The new method when issued will carry a new TAPPI number. He suggested that the present methods T 203 and T 230 should be brought up to date by im-

provements in the procedure which would not change the level of the results. He thought this should be done even though the new and much improved method was in the making. After suitable words of caution by Dr. Clark this plan was approved. Dr. Martin outlined points at which T 203 and T 230 should be revised in order to cut down the time required for completing the test and in general for making the methods more reliable.

Chain Length Distribution of Cellulose. Dr. Martin reported on the activities of the ACS Subcommittee on Molecular Weight Distribution. The limitations of the proposed method were outlined. It was suggested that the nitration of cellulose should be considered as well standardized and that the viscosity method for determining the molecular weight of the fraction should also be considered as reasonably well worked out. The part of the method which will need most improvement is generally admitted to be the fractionation step. Dr. Broughton described his interest in the problem of chain length distribution and the committee discussed the possibility of having Dr. Broughton carry on work under a TAPPI grant. The chairman was requested to consult with Dr. Broughton and take whatever steps are necessary to get work started on a chain length distribution method suitable for TAPPI.

A Suggested Method for Determining the Accessibility of Pulp. The subject of the accessible and nonaccessible fraction (crystalline and noncrystalline) of cellulose was discussed by the committee. Dr. Broughton described experiments he has done along this line. The committee requested that Dr. Broughton write up a method for the accessibility of pulp which would be based largely on the work of Nickerson using anhydrous formic acid. The committee decided to propose this as a suggested method to draw comment from interested workers in the TAPPI organization.

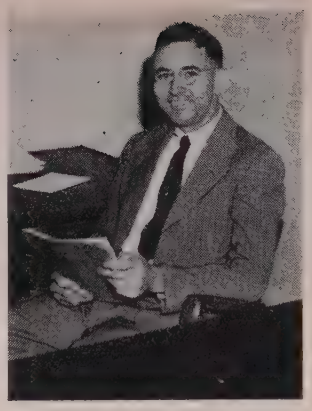
III. Standards for Paper

Pentosans in Paper, T 450. After considerable discussion it was decided to make slight revisions in T 450 to bring it into line with T 223. It was also suggested to actually make it a part of T 223 or refer back T 223 for the actual procedure for the determination of pentosans. The standard was turned over to Dr. McColley who will revise the procedure along the lines discussed by the committee.

Moisture in Paper by the Toluene Method. At present there is no TAPPI method for moisture in paper using the toluene technique. The Chemical Methods Committee thought it advisable to include this procedure for special papers which contain paraffin, varnish, and other materials which would be somewhat volatile at 110°C. and for which the regular oven method is not suitable. Dr. Broughton submitted a method based upon a British method [*Paper Maker and British Paper Trade J.* 119: 32–40 (1950)] as well as the ACS moisture Symposium article [*Anal. Chem.* 23, 1062 (1951)]. The precision of the procedure was compared with that of the newly revised oven methods for paper. The committee decided to submit the new toluene standard for balloting.

Starch in Paper, T 419 m-45. Considerable dissatisfaction was expressed with the present method for starch in paper. The hydrolysis using saliva was criticized as well as the method for determining the starch once it is removed from the paper. Dr. Browning, who could not attend the meeting of the committee, sent his comments in on this procedure stating that an improved method is in the process of development and could be presented to the committee probably at the February meeting. Dr. Browning was asked to serve as a subcommittee chairman and to bring recommendations for an improved starch in paper method.

Water-Soluble Sulphate and Chloride in Paper and Pulp, T 229 and T 468. There was considerable discussion of these standards by the committee. The discussion indicated that water-soluble salts in paper were of most interest to the electrical industry. General Electric, Westinghouse, and other



L. E. Simerl, Marathon Corp., Chairman, Packaging Materials Testing Committee

A. S. O'Brien, Eastman Kodak Co., Chairman, Chemical Methods Committee

concerns will be consulted by the committee to determine the method suitable for their special needs. Most of the industry, however, needs methods for total sulphate and chloride. Such methods should be described separately and given separate TAPPI numbers rather than lumping together in one standard.

Two or three other methods for constituents in paper were discussed briefly by the committee. The first was a "Zinc and Cadmium in Paper" procedure for which the ASTM has a method. There did not appear to be any great need for this procedure but a subcommittee was appointed to check into the matter and propose a standard. T 421, Qualitative Analysis of Mineral Filler and Mineral Coatings was discussed. The present 1944 revision seems to be adequate and the chairman was asked to appoint a subcommittee to make minor changes and to bring the method up to date.

Titanium Pigments in Paper, T 439. Mr. Willets of the Titanium Pigment Corp. offered to solicit the advice of his laboratories on the acceptability of T 439 in its present condition.

ASTM also has published a method for "Water-Soluble Matter in Paper." Although the need for this method does not appear great it was decided to have a subcommittee study the ASTM Procedure and submit a similar TAPPI Standard.

A. S. O'Brien, *Chairman*
Chemical Methods Committee
Eastman Kodak Co., Rochester, N. Y.

Packaging Materials Testing Committee

The joint TAPPI-Packaging Institute Packaging Materials Testing Committee met in two 3-hr. sessions on Sept. 11, 1952, at the State College of Forestry, Syracuse, N. Y. Eleven committee members or their representatives were present from a present membership list of 22, and the total attendance was 35. The following projects were discussed.

No. 277, Water Vapor Permeability at 0°F. The proposed method is now in the final stages of revision, having been forwarded by the Testing Division to the Standards Committee. The procedure was reviewed for the benefit of those not familiar with the development phases, and some minor suggestions accepted which will be incorporated in the final draft.

No. 387, Water Vapor Permeability at 100°F. and 90% R. H. Charles Southwick, chairman of the subcommittee handling this project, could not be present. The necessity for revision was reviewed, and the general reproducibility of the test was discussed. It was pointed out by a representative of a glassine mill that eight glassine manufacturers check semiannually within 10% tolerance. This test is run under the auspices of the Glassine Association, and they feel that the General Foods cabinets are acceptable for this purpose. Arthu

Landrock of MIT and Milton Yezek of General Foods at Battle Creek volunteered to assist the work of the subcommittee.

In the discussion of the 100°F., 90% R. H. W.V.P. procedure, an article by C. R. Oswin, "A Rapid Water Vapor Permeability Comparator," published in *Chemistry and Industry*, June 9, 1951, was reviewed. This procedure involves the use of radioactive hydrogen (tritium) and then sweeping the water vapor through a slow type counter. Copies of the article were sent to the subcommittee on water vapor permeability, and it is recommended that a study be made of additional work which may have been done since the publishing of this article. It was generally agreed that the equilibrium time as reported in the article, ranging from 3 to 10 min., was probably unrealistic in view of the known time required for cellulosic materials to come to equilibrium with a given relative humidity.

Creasing of Paper for W.V.P. Tests, T 465 sm-45. There was some discussion of the present TAPPI Standard Creasing Method, T 465. A discussion of the severity of the test led to the general conclusion that those companies interested in high quality, multilayer laminations using synthetic resins and other very tough material believe that the present creasing procedure is probably not severe enough to duplicate actual rough handling such as occurs in the military packaging field. On the other hand, there is some evidence to support the belief that the present TAPPI standard creasing procedure is too severe for such materials as plain waxed paper, constituting a considerable tonnage, used as innerwraps and overwraps for cartons. It may be desirable to set up two degrees of severity in the creasing procedure, one for materials which are expected to stand relatively little rough handling, and another procedure.

On the creasing procedure, Milton Yezek of General Foods and Lois Hans of the Quartermaster Research and Development Laboratory at Philadelphia were volunteers to assist on committee work.

No. 328, Flavor and Odor Transfer. Arthur Landrock, chairman of the subcommittee, described the present status of two procedures, one involving organoleptic methods and the other the methyl furoate analysis. These procedures are now in rough draft stages within the Testing Division, and will be forwarded as fast as possible to the Standards Committee.

No. 331, Heat Sealability. This is a rather complicated subject which has been discussed at some length at all of the previous committee meetings, and is closely allied to a similar project in the joint TAPPI-ASTM Wax Testing Committee. There was a discussion of the factors involved in setting up such a test procedure for a specific type of material, as follows:

1. Determine optimum temperature, time, and pressure for the given heat sealing coating.
2. Prepare seals at the optimum conditions found above.
3. Determine the seal strength in grams per centimeter width or other units, on any one of several available tensile testing machines, using a direct 180° pull.

Lois Hans of the Quartermaster Research and Development Group stated that a heat sealing machine was available with control over the three factors (Packaging Industries Corp., Montclair, N. J.).

Among tensile testers available, the Palo-Myers instrument manufactured by Palo-Myers of 81 Reade St., New York 7, N. Y., has found some acceptance by several companies interested in the range from 0 to 200 grams. A tentative procedure was published by the Packaging Institute in *Modern Packaging*, September, 1946, pages 150–51, 180–82. There have been some significant developments since that time, and if the need exists, a tentative procedure can be issued by TAPPI at this time. An expression of interest is required from TAPPI membership.

No. 357, Water Vapor Permeability of Completed Packages. Charles Woodcock, chairman of the subcommittee, could not be present. Two procedures, one using a standard size package with calcium chloride as desiccant, and a second procedure which is more general, allowing comparison of any size package, with the product being packaged as the test material, and including rough handling, are in rough draft stage within the committee. These procedures will be forwarded to the Standards Committee as soon as possible. The discussion of these procedures at the meeting indicated considerable interest.

No. 388, Insect Resistance. Howard Gardner, chairman of the subcommittee, was unable to be present, but was represented by Dr. David Edwards. This procedure is in final draft stages within the committee, and will be forwarded to the Standards Committee as soon as possible. There was considerable discussion of the need for a standard procedure in the industry, with representatives of several companies present who are now manufacturing insectproof materials.

New Projects

Under the possible new projects for the committee, a standard test for mold growth susceptibility was suggested. This procedure is of primary importance to the Armed Forces Procurement Agencies, and it was indicated that all Ordnance wrap would require such a treatment and a test would be required by the specification. It is believed that there is sufficient background available, particularly in the Armed Forces Laboratories, for such a standard test procedure, and this can be handled by TAPPI if there is sufficient demand. The possibility that this would be in the jurisdiction of the Microbiological Committee was suggested.

The problem of odor of packaging materials was also brought up. This procedure, or general collection of procedures adaptable to several types of packaging materials, was reviewed for the benefit of those unfamiliar with the committee work to date. A tentative method now exists. There is apparently sufficient interest to justify rewriting the procedure in TAPPI standard form, and forwarding through channels for approval.

One additional request for a standard concerned the gluing of boxboard. Specifically, a sizing test which will correlate with the absorption of adhesive in the gluing operation was required. In the general discussion, it was suggested that the water drop method now generally used be replaced by a wet cellulose sponge test, in which several square inches of board are subjected to moisture for a definite period of time, and the moisture pickup by the board determined by weighing. It was pointed out that the Packaging Institute are very much interested in adhesive problems, and that there is a forum on adhesive use at the meeting of the Packaging Institute on Oct. 20–22, 1952, in New York City.

The next two meetings of the Packaging Materials Testing Committee will be on Monday afternoon, October 20, at 2:00 p.m. at the Hotel Commodore, and at the Annual Meeting of the Technical Association at the Commodore Hotel, New York, at 2:00 p.m. Monday, February 16.

L. E. Simerl, *Chairman*
Packaging Materials Testing Committee
Marathon Corp., Menasha, Wis.

Paper Testing

The meetings of the Paper Testing Committee held at Syracuse were very well attended, not only by committee members, but also by other interested persons. Because of the excellent attendance and complete cooperation from those attending, we were able to accomplish much work. I think that the best way to make this report is to follow the planned agenda and report the action taken.

1. *No. 320, Blood Resistance of Butcher Wrapping Paper.* Prior to the meeting, Jack Ward of Riegel Paper Corp. had prepared a tentative draft of a test method for determining

the blood resistance of butcher wrapping paper. This tentative draft was gone over with a fine tooth comb by everyone attending, and with their able assistance, we were able to write a suitable method which when rewritten and edited, will be submitted to the committee members for adoption by TAPPI as a suggested method.

2. *No. 423, Measurement of Pin Holes in Glassine and Greaseproof Papers.* Prior to the meeting, I had prepared a draft for a method for Measurement of Pin Holes in Glassine and Greaseproof Papers, fashioning it to a great deal after an adopted method by ASTM. This draft was worked over and corrected to the extent that when rewritten and edited, will be in shape to be presented to the committee members for vote to adopt it by TAPPI as a suggested method.

3. *Discussion of TAPPI Method T 469 sm-45, Flexural Resistance and Deflection of Fiberboard (for possible advancing from a suggested method to a tentative method).* There were no comments or criticism of this test method, and, therefore, it will be submitted to the committee members for vote to advance it from a suggested method to a tentative method. The only remarks made were to bring up the question as to why this method was under the jurisdiction of the Paper Testing Committee, the method dealing entirely with fiberboards such as binder board, insulating board, wallboard, and hard-pressed structural board.

4. *Discussion of Method T 470 m-47, Edge Tearing Resistance of Paper (French Method) (for possible advancing this method from a tentative to an official standard).* There were no major criticisms or suggested changes to this method, although committee member, Mr. Kantrowitz, is going to check with the joint TAPPI-ASTM Committee for minor revisions. After receiving his report, this method will be submitted to the committee members for vote to advance this method from a tentative to an official method.

5. *Review of Test Methods.* (a) *Thickness and Density of Paper, T 411 m-44:* There was an active discussion about this test method and there were several good suggestions for minor revisions, such as deleting from the paragraph on "Definitions" the words "in thousandths of an inch," which are in parentheses and actually meaningless because the test method calls for reporting results either in decimals of an inch or millimeters. It was also suggested that in the paragraph entitled "Test Specimen," the wording be revised to incorporate the fact that test samples shall be taken in accordance to TAPPI Standard T 400 m. It was further suggested that the first sentence of the paragraph under "Report" be changed to read "The thickness shall be reported in decimals of an inch to the nearest 0.0001 in. (0.002 mm.)," to "two significant figures together with maximum and minimum readings and number and size of the specimen sheets."

A lengthy discussion was held concerning the measurement of the thickness of thin papers, a subject not included in T 411 m-44. The U. S. Bureau of Standards has issued circular No. 532, "Measurement of the Thickness of Capacitor Paper," which is a very thorough study of the theory and practical solution of measuring the thickness of thin papers. This committee is going to look into the possibility of incorporating a specific method for measuring the thickness of lightweight papers into the present TAPPI method, basing the method on the conclusions drawn by the study as reported in the National Bureau of Standards Circular No. 532. This work will continue, and in all likelihood, will be a subject on the agenda for our next meeting.

(b) *Dirt in Paper T 437 m-43:* This method was placed on the agenda for discussion for possible corrections and advancement from a tentative to an official method. It was unanimously agreed that the paragraph "Apparatus" should be rewritten, bringing it up to date by deleting the "Note" and making a reference to the fact that the standard comparison chart required was printed in 1942 and is obtainable from the Secretary of the Association. The wording of the procedure was brought under quite some criticism, because of the reference to a 50-watt Mazda frosted bulb. For one thing, this is not a very scientific way of describing the necessary illumination and also 50-watt bulbs are no longer manufactured. It is the opinion of this committee that a much better approach to this subject would be to express the illumination in so many foot-candles and perhaps include an example of how that amount of illumination can be obtained. Therefore, this method is being sent to a subcommittee for further study and revision.

(c) The following test methods were reviewed for possible corrections or revisions, and will be submitted to the committee members for vote for appropriate action as indicated: *Alkali-Staining Resistance of Papers, T 440 m-42*—Already an official method, the committee will vote whether or not to retain this method as presently written. *Wire and Felt Sides of Paper, T 455 m-44*—This method will be put up to the committee members for vote for advancing from a suggested to a tentative method. *Printing-Ink Permeation of Papers (Castor-Oil Test), T 462 m-43*—This method will be submitted to the committee members for vote for possible advancement from a tentative to an official standard.

6. *Other Business.* There seemed to be general agreement that TAPPI Method T 433 m-44, Water Resistance of Paper and Paperboard (Dry Indicator Method), should be opened for possible revision along the lines already adopted by ASTM. This suggestion is being taken under advisement for possible subcommittee action.

DONALD H. NEWCOMB, *Chairman*
Paper Testing Committee
Riegel Paper Corp., New York, N. Y.

Wax Testing Committee

Section I—Strength Tests (Syracuse, N. Y., Sept. 10, 1952: J. W. PADGETT, *Chairman*, and D. R. CUSHMAN, *Secretary*) The minutes of the previous February meeting were read, and copies were distributed of a final report by Mr. Padgett summarizing the results of the second round-robin tensile strength tests. A detailed study and discussion of the report followed with conclusions in good agreement with those in the report. From an over-all standpoint, the Modified Dow Mold was shown to give the best results, but the Mellon Modification of the Perkins Mold was indicated as being only slightly poorer. Results using the Standard Perkins Mold showed a marked improvement over those reported in the first round-robin test, and it approached the precision of both the Modified Dow and Mellon Modification of the Perkins Mold. This improvement in precision probably is due to the suggested changes in the technique of preparing specimens incorporated in the method used in the last round robin.

The desirability of revising the section relating to mold design in the test procedure to be published "for information only" in ASTM Committee D-2 Standards on Petroleum Products, and as a suggested TAPPI Method, was discussed in some detail. This procedure calls for use of the Perkins Mold. However, in view of comments received from several committee members during the letter ballot on publication of the procedure, arrangements were made to add a footnote relative to the current status of the molds. The committee concurred that addition of this footnote clarified the situation and voted to approve addition of the footnote.

On the question of mold design, Mr. Padgett emphasized the need for further cooperative testing, with more laboratories participating and comparing the various molds, with the objective of adopting a standard mold design.

A discussion on the use of mold release compound followed and it was agreed that the latest method, involving the application of a thin film of mold release compound with a cloth, followed by wiping, should not affect the tensile value of paraffin waxes appreciably. Some results were undoubtedly

low because of the use of an excess of mold release compound. Esso Standard Oil and Standard Oil of Indiana are further investigating the effect of mold release compounds.

The effect of relative humidity at the time of pouring and during cooling was discussed, and it was decided to set up a program to study the variation of this factor to form a basis for setting up tolerances. Members with constant humidity rooms will use them; otherwise cabinets can be used for control of conditions during pouring and cooling of the specimens. After the wax has solidified and cooled to the test temperature, the specimens will be conditioned in the normal manner, including immersion in water for 15 min. prior to testing.

J. Clark of TAPPI suggested the desirability of selecting one manufacturer to make the molds, and it was decided that in the minutes a statement be included naming a manufacturer from whom molds conforming to the specifications could be obtained. In accordance with the decision of the committee, molds can be obtained from the following manufacturer: W. H. Schrader, Instruments and Test Apparatus, 3459 Queen Lane, Philadelphia 29, Pa.

On the question of standard waxes for calibration, I. E. Manning of Esso Standard Oil, Bayonne Laboratories, Bayonne, N. J., agreed to supply these waxes, and all standards should be obtained directly from him.

The need for maintenance and calibration procedures was emphasized and D. R. Cushman of Socony-Vacuum Oil Co. agreed to work out tentative procedures, starting with the Tinius Olsen tester.

The work on bending tests is to be continued, with F. H. MacLaren (or P. J. Gianacakes), Standard Oil of Indiana, carrying out this phase of the program.

Section II—Flow Tests (Syracuse, N. Y., Sept. 12, 1952, H. Schindler, *Chairman*): Section II has completed round-robin penetration tests on six wax samples (paraffin and microcrystalline wax) at 77, 90, 100, 110, and 120°F. using both the standard needle of ASTM D-5 (asphalt penetration) and a special needle designed by Standard Oil Development Co. The results confirm previous findings concerning the precision of the proposed method for determining the penetration of petroleum waxes. It was agreed that the precision is satisfactory for penetrations up to 50—regardless at what temperature they have been determined—and the method will accordingly be submitted for publication as a "suggested TAPPI Method." After complete statistical evaluation, which will furnish data on the limits of precision and repeatability among laboratories, the method will be submitted to ASTM Committee D-2 for "publication for information."

Further cooperative tests will be run with the Abrahams Consistometer using the procedure of the Moore & Munger laboratory. California Research Corp. will submit hardness data on the cooperative samples obtained by a special method used by that company and Western Waxed Paper Co.

Section III—Stability Tests (Syracuse, N. Y., Sept. 11, 1952, G. P. Hinds, *Chairman*): Section III has continued work on the accelerated oxidation test using the rate of development of peroxides as a measure of wax stability. A "round-robin" test, conducted since the February, 1952, meeting, has indicated that the reproducibility of the test in any one laboratory is quite good, but that agreement between different laboratories is still unsatisfactory. Four waxes of varying stability have been tested, and each reporting laboratory evaluated them in the same order, although there were wide differences in the values obtained for any one wax. At this point the method is capable of clearly distinguishing between stable and unstable waxes, but results between laboratories are unreliable in borderline cases.

The committee felt that the results obtained to date justify further work on the peroxide method. It was believed, however, that all test variables had not as yet been sufficiently controlled. Consequently, the test procedure was modified somewhat, and the committee agreed to critically review the peroxide number method used at present. Modified methods for this test are to be circulated for comment, and a sample of oxidized wax is to be distributed by the chairman to be tested by the committee members. The peroxide number method will then be modified according to the suggestions received.

Another cooperative test will be carried out using the oxidation procedure and peroxide number method as modified. The committee felt that these changes should improve the reproducibility of the test between laboratories. Should it not do so, some other type of test will require investigation.

A few data are available on the Shaker method, a relatively simple technique which measures the oxygen absorbed by the material under test. The committee recommended that more data be obtained on this method for consideration, in case continued difficulties are encountered with the peroxide technique.

It was agreed that no publication of methods should be considered until test reproducibility between laboratories has been improved. It was hoped that by the February, 1953, meeting sufficient data will be available to proceed along this line.

Section V—Composition (Syracuse, N. Y., Sept. 12, 1952, J. B. Rather, Jr., *Chairman*): A meeting of Section V, Composition, of the TAPPI-ASTM Technical Committee on Petroleum Wax was held Friday, Sept. 12, 1952, in Syracuse, N. Y. The following business was transacted:

1. In view of the fact that Section V had not met in conjunction with the TAPPI Group for some time, the chairman reviewed the group's past activities over the last year, and distributed to those present a memorandum dated June 20, 1952, entitled "Summary of Current Cooperative Work on Determination of Oil in Petroleum Waxes." Briefly, the work of the Section indicates that there is a good correlation between oil content results obtained using methyl ethyl ketone at −25°F. and ethylene dichloride at 0°F. No valid objections have been found to the use of methyl ethyl ketone as the solvent for microcrystalline wax and the reproducibility of the oil content results using this material is excellent. In fact, reproducibility was actually found to be even better than that presently stipulated in D 721–51 T.

2. The group discussed the proposed work agreed to at the June 24, 1952, meeting of Section V having to do with the need for better precision data on oil content values for paraffin waxes. In that connection, the chairman announced that his laboratory has prepared the following synthetic samples representing paraffin waxes of various oil content levels. The samples in question were prepared by blending a low oil content paraffin wax with a 100-sec. paraffin oil. The waxes are listed below:

Sample	*Oil added, %*	*Oil found (one test), %*
1	..	0.02
2	0.5	0.39
3	1.0	0.93
4	5.0	4.4
5	10.0	8.9
6	15.0	14.0

All of the above samples were made from sample 1, which contains no added oil. It was agreed that these samples would be satisfactory for repeatability and reproducibility work. Accordingly, the chairman was instructed to proceed to distribute them to the members of the Section with the view of obtaining the data some time before the February, 1953, meeting of ASTM Committee D-2.

3. A motion was passed unanimously to replace Method D 721–51 T with the Proposed Method of Test for Oil Content of Petroleum Wax, which appears as Appendix IV of the 1951 report of ASTM Committee D-2 on page 714 of the ASTM Standards on Petroleum Products and Lubricants. This action on the part of Section V will be implemented by letter ballot of the TAPPI-ASTM Technical Committee on Petroleum Wax (Technical Committee M). This letter

ballot will be prepared shortly and the formal recommendation with respect to the change will be made to Committee D-2 in February, 1953. In connection with this action, it is understood that the aforementioned procedure will be recommended to TAPPI for similar publication as a tentative standard. Later on, it is anticipated that the procedure will be elevated to a standard.

4. The group's attention was called to a letter written by the chairman under date of Sept. 9, 1952, to Sidney Minchin, Chairman, Subcommittee 8—Petroleum Waxes, of the Institute of Petroleum, presenting answers to various questions relating to differences in ASTM and I.B. and I.P. test procedures on wax. The need for international cooperation of this character was emphasized.

5. There was some discussion relative to which section of the TAPPI-ASTM Committee handled melting point work, i.e., Section II, Flow Test or Section V, Composition. This matter subsequently was referred to the Advisory Section and a decision was reached to consider melting point work to be within the province of Section V, Composition. With respect to possible work on melting point, two matters were discussed: First, it is understood that J. W. Padgett of Moore and Munger, has available a melting point method which he is agreeable to make available to Section V for study. It was agreed that consideration would be given to investigating Mr. Padgett's procedure. Secondly, a number of opinions were expressed to the effect that it might be desirable to extend the scope of Method D 938–49, "Congealing Point of Pharmaceutical Petrolatums," to include petrolatums and microcrystalline wax. At the present time, the scope of D 938–49 specifically confines the method to pharmaceutical petrolatums referring to Method D 127 for the latter waxes. It was decided that the chairman should draft a questionnaire to be sent to the Joint Committee for the purpose of ascertaining present views on the matter. It was brought out that D 938–49 had been confined to pharmaceutical petrolatums to avoid possible difficulties in Federal and commercial specifications which now, rather generally, specify D-127.

6. It was decided that, in general, it would be desirable for Section V to meet with the TAPPI group about once a year.

Section VI—Blocking Point (Syracuse, N. Y., Sept. 10, 1952, H. F. Hitchcox, *Chairman*): Results of cooperative blocking tests on 18 waxes were reviewed. Considering that these were the first round-robin tests by the new temperature gradient technique, the agreement between laboratories was good. With seven companies reporting, the blocking data for most waxes agreed within 3 to 5°F. from five or six of the laboratories. Results by four companies by the mercury method agreed within 5°F. for about 80% of the waxes; three waxes showed 10 or 15°F. differences in reported values.

The inaccuracies in the present test were believed due to (1) improper calibration of thermocouples and the blocking plate, (2) differences between laboratories in the conception of the blocking point, and (3) to some extent, conditions of coating and aging the test strips. Another round-robin test will be made with the same waxes. It was agreed that each laboratory would recalibrate its equipment. Blocking tests will be run with both air-cooled and water-chilled test strips and the strips will be aged between 25 and 30 hr. before placing them on the blocking plate. It was further agreed that, for these tests at least, the blocking point would be judged as the point at which 50% of the width of the strip showed picking or a definitely disrupted surface. Spotting without film disruption, or loss of gloss is not to be interpreted as blocking.

No attempt was made to judge the relative merit of the temperature gradient versus the mercury method. It is hoped that such a comparison may be drawn at the February meeting.

Section VII—Sealing and Laminating (Syracuse, N. Y., Sept. 11, 1952, G. C. Borden, Jr., *Chairman*, and J. H. Kirk, *Secretary*): The Method for Seal Strength of Paraffin Waxes drawn up for publication in *Tappi* as information only was discussed in detail. Two minor changes in the wording of the procedure were approved by the committee: (1) the width of the specimen to be sealed was set at 5 in., thus making a definite width to be used with the 200-gram load and (2) the use of "grams per inch" as the unit in reporting results rather than "grams per centimeter" as originally drawn up.

George Mueller questioned if the publication of the method was justified at this time because sufficient data were not available to establish the method as superior to the Socony-Vacuum 90° separation method.

After considerable discussion, a motion was approved to go ahead and publish the corrected method in *Tappi* as a suggested method with a qualifying paragraph explaining that the committee believes the method is the best available and that further work is being done especially regarding the relative merits of the 90 and 180° angle of separation of the sheets.

The second round-robin of tests as set up at the February meeting was discussed. In addition to the low seal strength wax sent out by Mr. Turner, an intermediate and high seal strength wax will be sent out by Mr. Padgett for additional tests.

Little or no progress has been made with the microwax lamination phase of the program. It was agreed to go ahead and try to apply the procedure for seal strength of paraffin to the microwax using glassine in place of the bread wrap. Based on the experience gained from the experimental work, a more definite program can be set up at the February meeting.

Considerable interest was shown in the use of a water quench for cooling the sealed specimens. It was agreed to investigate the water quench for possible inclusion in the test procedure at a later date.

Section VIII—Scuff and Gloss (Syracuse, N. Y., Sept. 10, 1952, G. P. Mueller, *Chairman*): *Gloss*—Past history of work done on gloss was briefly summarized including the virtual elimination of all fixed angle glossmeters as capable of measuring the gloss of high-gloss waxed papers accurately. Mr. Gianacakes then presented a brief report on Standard of Indiana work using the Hunter Goniophotometer. Their work shows that:

1. The 45, 60, 75, and 20° fixed angle meters do not give correlation with visual ratings.

2. Variation in aperture size of the pickup cell had no appreciable effect on gloss values.

3. Goniophotometric curves can be correlated with visual ratings based on the peakedness constant or the shape and area of the curves.

No reply has yet been received from the TAPPI Optical Properties Committee in answer to our request to devote some fundamental study to this problem. Further work will await this reply.

Scuff—Data were presented by Mr. Mueller showing results of scuff tests performed on the Marathon Tester using, as scuffing surfaces, striated steel blocks, ground glass, and desiccated tissue paper. Results indicated that good correlation exists among these surfaces, with highest values being obtained with the paper and lowest with the glass surface. Since the values obtained using steel were very near those obtained using paper, and since the steel surface is more reproducible, further work will be done using the steel. Marathon will supply steel blocks to all holders of Marathon Testers, and testers will be furnished to additional laboratories to obtain additional data.

In addition, further work will be done using Dr. Mazee's method and Fiberboard's method. Waxed sheets will be supplied to the testing laboratories by the chairman.

W. J. Yates, *Secretary*
TAPPI-ASTM Technical Committee on Petroleum Wax
Shell Oil Co., New York, N. Y.

LOCAL SECTION ACTIVITIES

Reports of Meetings, Personnel and Events

Southeastern

The Southeastern Pulp and Paper Society became the Southeastern Section of the Technical Association of the Pulp and Paper Industry at its Charter Meeting at the King and Prince Hotel, St. Simons Island, Ga., on Sept. 19–20, 1952. About 150 individuals were present at the dinner meeting on Sept. 19.

The meeting was opened by Glenn C. Kimble, Union Bag & Paper Corp., Savannah, Ga., Chairman of the new section. E. J. Gaynor, 3rd, President of the Brunswick Pulp and Paper Co., Brunswick, Ga., extended a welcome to those present. Reavis C. Sproull, Director of the Herty Research Laboratory, Savannah, Ga., presided during the formal speaking program. C. E. Tyler, Chief Chemist of the Hercules Powder Co., Brunswick, Ga., extended an invitation to all to make the tour scheduled for the 20th of the Naval Stores plant of his company. He described the various processes that would be seen at the plant. Early in the meeting an interesting interlude featured the good wishes of the members to Cap Youngchild on the occasion of his 70th birthday.

The officers of the Southeastern Section are as follows:

Chairman—Glenn C. Kimble, Union Bag & Paper Co., Savannah, Ga.
Vice-Chairman—Malcolm B. Pineo, Brunswick Pulp & Paper Co., Brunswick, Ga.
Treasurer—George E. Scofield, Rayonier, Inc., Fernandina, Fla.
Treasurer—George E. Scofield, Rayonier, Inc., Fernandina, Fla.
Recording Secretary—Wilson F. Brown, University of Florida, Gainesville, Fla.
Corresponding Secretary—James E. Overall, Union Bag & Paper Corp., Savannah, Ga.

The Charter Address was given by R. G. Macdonald, Secretary-Treasurer of the Technical Association of the Pulp and Paper Industry, New York, N. Y. Mr. Macdonald's address follows:

TAPPI in the Southeast

R. G. Macdonald

The present occasion is an event of historical importance since it symbolizes the great growth and importance of the pulp and paper industry in the Southeast.

The period of influence of the Technical Association in this region covers a period of 20 years. Just 20 years ago the industry was aware of the effort being made by the late Charles H. Herty. Dr. Herty, with the aid of the Chemical Foundation, had established his now famous pulp and paper laboratory at Savannah.

In 1932 at the invitation of Dr. Herty and several associated interests, the Technical Association sponsored a pleasant barnstorming trip. The Savannah Liner *City of Montgomery* was chartered and about 150 individuals traveled from New York to Savannah. At Savannah the Association held a joint meeting with the Georgia Forestry Association to discuss prospects for a new pulp and paper industry in Georgia.

By 1937 the true possibilities of the South as a pulp and paper region were known. In those days the Association held Fall meetings of national status. Savannah was selected for the annual event. Under the tremendous effort of D. G. and Edna Moon of the Union Bag & Paper Co. and their associates a wonderful meeting was held. The housing problem was prodigious since more than 1200 individuals arrived and were accommodated, somehow. I do not recall the technical program but a hundred events of social significance are recalled. It was a carnival if there ever was one.

It created a local enthusiasm that was reflected in the folfowing years by the Southern Pulp and Paper Festival and the Coastal Plain Paper Festival, the first challenging the great Mardi Gras at New Orleans. Mr. and Mrs. Moon put over the first festival, an extravaganza nonpareil. I understand that the fussing and feuding over the crown jewels of the Paper Festival Queen destroyed the courage of the Savannah Chamber of Commerce. There was no third festival.

Although there were relatively few technically trained men in the mills there was a desire on the part of these men to organize a local society. By this time mills had been built at Jacksonville, Fernandina, Brunswick, Savannah, and Charleston. The spirit of the times led to the establishment of an organization called the South Atlantic Pulp and Paper Society which held a few meetings. It was a little premature but provided a background of experience that was probably in the minds of your present organization. I do not know of any TAPPI Local Section that approached its organization problem as carefully as did the Southeastern Pulp and Paper Society. As a result it has been built on a firm foundation, having obtained as it has the wholehearted endorsement of its company managements.

Its appeal to the company management has been based on a seriousness of purpose. To date this confidence has been justified and the present meeting reflects the real purpose of the Southeastern Section to provide you with programs featured by mature consideration of the major technical problems of the mills in this region.

R. G. Macdonald passing charter and gavel to Glenn C. Kimble

R. G. Macdonald, Secretary-Treasurer, Technical Association of the Pulp and Paper Industry, New York, N. Y.

J. E. Overall, P. B. Borlew, James Clark, C. E. Libby, Claude Rucker, Kenneth Ross, Thomas Hardwick, John Morris, B. B. Boyd, and B. A. Towers

When I use the word technical I mean the presentation of information acquired by expert experimentation, observation, and the interpretation of such data in terms of their practical application to the processes of pulp and papermaking so that they will be most efficient and economically sound.

In addition to the interchange of ideas on professional subjects that will result from your effort there is another objective for which local sections exist. This objective is the development of leadership qualities, especially among your younger members. The opportunities for self-expression are almost unlimited. In addition to the benefit of maintaining an organization such as yours there is the opportunity for participation for everyone who attends your meetings.

Last week I had dinner with a highly competent Association member. In the course of our conversation he reminisced a little about his past experience. In doing this he mentioned that he recalled the occasion clearly when the top executive of his company was observing him and others to decide who should be given an unusual opportunity to move into a management position. It is in retrospect that he realized that he had missed a golden opportunity because he was tongue-tied on the occasion and did not produce the impression that would have instilled confidence on the part of his superior that he could handle a job where clear thinking and articulate expression were of considerable importance.

To quote: "There comes a time in the tide of affairs of man, which if grasped at the crest, will lead to glory."

In my years of travel through these southern states I do not recall meeting very many individuals who were inarticulate or self-effacing. Being a native of the New England States I can assure you that this personality contrast between many individuals in these two regions is startlingly noticeable.

For this reason I will present to your chairman this evening a gavel large enough and strong enough to restrain your discussions to the extent that he may consider desirable for the good of the order.

However, I wish to recommend to your officers and your program committee that your meeting programs be arranged on the basis of permitting and urging you to take an active part. I hope that a large proportion of you will be members of panels and will enter into the discussions. You know each other quite well and, in time, will know each other better. It you are called upon to give your advice please give it to the extent that you are able and willing to do so.

If you do your part, as I feel sure that you will, there is no reason why the Southeastern Section cannot be one of the leading sections of the Association. You are arriving late but you are arriving under auspicious conditions. Strangely enough, the first of the TAPPI sections was established on the Pacific Coast in 1929, and not in one of the older papermaking regions. It was established by pioneers engaged in mastering the pulp-making potentials of a vast industry frontier of big trees and limitless forests. Your speaker tonight, Ray Hatch, was one of those pioneers. He cannot fail to inspire you. As a TAPPI Medallist he is a member of the exclusive circle of the industry's great men.

Although it is 23 years later than 1929, when the Pacific Section was formed, you are arriving at a time when great things are expected of you. You are employed in a gigantic and complex industry. You need to know much more than was known in 1929 and there never existed a time when able men were more needed.

This Section, with your united effort, can make real contributions to the technical advancement of the pulp and paper industry. You can sponsor programs that will have an international audience through the medium of your Association publication *Tappi* and the trade journals, especially your own private *Pulp and Paper Manufacturer*. You can be sure that Vince Waters will be in there pitching for you all of the time. Vince is a graduate of the Technical Association staff, a founding father of the Ohio Section, and one of TAPPI's genuine boosters in the Deep South.

You, too, can be proud of your membership in the Southeastern Section and the Technical Association of the Pulp and Paper Industry. It can be the kind of organization that you are willing to make it. Get into the action; fight for it, struggle for it, work for it. The results will be worth while. When the Section has had sufficient experience it will have jobs to do for its parent Association.

Mr. Chairman, the petition of the members of the Association associated with you in the Southeastern Pulp and Paper Society was received by the Executive Committee of the Association and your request that a charter be granted to your group was unanimously approved.

I, therefore, present you with this symbol of a charter establishing the Southeastern Section of the Technical Association of the Pulp and Paper Industry.

Here also, is your gavel of authority as Chairman of the Section and on behalf of the Association's Executive Committee I present the Section with a banner. As it stands before you at your meetings I trust that it will be observed with growing pride, symbolizing as it does the service of your members who are dedicated to the task of contributing to the welfare of their fellow men through the manufacture of better products from our beautiful forests.

One of the principal addresses was given by Raymond S. Hatch, Research Director of the Hudson Pulp & Paper Co., Palatka, Fla. This address, "Research in the Pulp and Paper Industry" follows:

C. E. Tyler, W. A. Wier, L. A. Thompson, T. P. Pace, Leroy Clark, Jay McClane, A. S. Baker, and G. H. Cook, Jr.

Research in the Pulp and Paper Industry

Raymond S. Hatch

On the occasion of the establishment of a new section of TAPPI in the Southeast, it seems to be fitting to discuss the role of research in our industry.

World War I demonstrated conclusively how much this country depended upon foreign scientific advancement for many of the products needed both for offense and defense. Our industrialists, therefore, began to turn to research as a means of keeping pace with scientific advancement throughout the world. This progress was well under way when we were confronted with the emergencies of World War II. It is hardly necessary for me to dwell upon the contribution of research in this country to the successful conclusion of this second great war. The lessons learned in its trying years are evident on every hand in the growth of great industrial research laboratories in almost every field of endeavor.

Up until comparatively few years ago, our great petroleum industry was devoted entirely to the production of liquid fuels and lubricants. But, as a result of research, the petroleum industry is today producing many useful chemical compounds in tank car lots which, a few years ago, were only laboratory curiosities.

I cite the petroleum industry because of its youth. Our industry, on the other hand, has a much longer history, dating back almost to the beginning of the Christian era.

Our raw material is a renewable resource. As long as the sun shines, we are assured of a supply of cellulose from our fields and forests. Cellulose, however, is not the only product resulting from the captured energy of the sun. In productive capacity, we are now approaching a cellulose production of 20 million tons a year, but this huge production represents less than half of the growth which we are harvesting annually. That portion of vegetation which is greater than our cellulose production is so-called waste. The kraft industry was compelled from its inception to recover the relatively costly chemicals used for the production of sulphate pulp. Since this recovery process required the burning of the organic material present in the waste liquor, it was only natural for the industry to utilize the heat of this combustion for the generation of steam and power, one of the essential raw materials for pulp production.

For many years, the sulphite industry made no attempt to utilize sulphite waste liquor because of the low cost of the chemicals used for the production of sulphite pulp. This waste imposed a serious pollution load on our streams which compelled the industry to investigate means of doing away

Raymond S. Hatch, Acting Research Director, Hudson Pulp & Paper Co., Palatka, Florida.

V. F. Waters, J. E. Reese, M. R. Roberts, R. S. Hatch, and Floyd Diephius

R. S. Hatch addressing the Charter Meeting of the Southeastern TAPPI

with this pollution. Added to the pollution problem has been the increasing cost of fuel and the developing scarcity of sulphur. Research has found a way to utilize the organic waste as fuel and to at least recover a large portion of the sulphur formerly wasted. As long as calcium is used as a base for the production of cooking acids, little hope for its recovery can be expected. By changing to a magnesia base, research has demonstrated that both sulphur and magnesia may be recovered. The use of magnesia as a base is, of course, only economically sound where relatively low-cost, pure magnesium oxide is available. By turning to ammonia as a base, the sulphur may be recovered but the ammonia is decomposed in the recovery process.

In the Scandinavian countries, where pure magnesia is not available, and where ammonia is more expensive than in this country, there is increasing use of the evaporated waste liquor as fuel to replace the high-cost fuels in those countries. The calcium base is lost as calcium sulphate and only insignificant amounts of sulphur may be recovered. However, the fuel value of the organic waste makes the process economically sound.

Let us now consider the nature of the organic material in the waste liquor. As you all know, this consists largely of lignin modified either by the sulphate or the sulphite process. In the sulphite process, we have in addition to modified lignin, both pentose and hexose sugars. The hexose sugars may be fermented to alcohol. The production of alcohol from sulphite waste liquor is economically sound as long as the price of alcohol remains at a relatively high level. Sulphite alcohol is, however, theatened by the increased production of relatively cheap synthetic alcohol by the petroleum industry.

It is also possible to produce butanol and acetone by proper fermentation of the sugars in waste sulphite liquor. In addition to the products mentioned, it is possible to utilize the sugars present in the waste liquor for the production of food yeast. This, as you know, is being done in one of our midwestern mills.

The conversion of the carbohydrates present in sulphite waste liquor does not utilize the lignin. Part of this lignin may be converted into vanillin and vanillin derivatives. But this utilizes a very small portion only of the total lignin available and the market for these products is limited.

The sugars extracted from the wood by the sulphate process are converted into acids by the action of the strongly

R. M. Hoyt, Owen Hunter, L. C. Thayer, Roy Homans, W. C. Chapman, John Baber, R. T. Hudson, R. C. Sproull, J. H. Oliver, and Victor Nelson

alkaline liquor and thus are not available for the production of alcohols or other products of fermentation.

As far as lignin is concerned, many uses have been suggested for this material, but so far, these uses have been small compared with the total lignin waste. Research in our industry should take as an example the strides made by the petroleum industry and by the intensive efforts made in recent years to convert coal into liquid fuels and valuable chemical products. After all, coal is but a metamorphosed form of lignin grown millions of years ago in our coniferous forests, and research should find a way to convert this renewable basic material into either liquid fuels or chemical compounds more valuable than the use of lignin as a fuel.

During the last twenty years, there has been considerable interest in the conversion of wood waste into alcohol or sugars through acid hydrolysis of the cellulose present in the wood waste. This method of approach is in direct competition with the production of sugar from annual crops, such as sugar cane sorghum, and sugar beets. Furthermore, the cellulose is destroyed and the lignin is so modified as to be of little use except as fuel. Wood waste in the lumber industry is enormous. But much of this wood can be converted into cellulose pulp more valuable than the conversion of the cellulose to sugars. This has been fully realized on the West Coast and today there is increasing use of wood waste for the production of high-grade pulp, thus effecting a material conservation of our wood supply.

We do not have to go back very many years to recall when the huge waste burners were a familiar sight in the lumber industry. The smoke and cinders from these waste burners filled the air around the sawmills. Today, it is difficult to find a waste burner in operation. Furthermore, as a result of research, there is less hogged wood fuel available for the generation of steam and power. Cellulose which may be obtained from wood waste, formerly recklessly burned, has become a valuable article of commerce.

Research may be divided into two broad classifications. Fundamental research is the search for scientific proof regardless of its practical use. Applied research is a broader field in some respects in that it takes the results of fundamental research and applies it to industrial process. Not only does applied research utilize the findings of fundamental research, but it also calls in the skill of the engineer, the forester, and the agriculturalist to combine their efforts in the production of better products at lower costs and to see that our supply of raw materials is not interrupted by overexploitation of our natural resources.

There is still ample room for fundamental research in the pulp and paper industry. I have been quite free in the use of the word "lignin," but what is lignin? We know something about some of the building stones of the lignin molecule, but we do not know the whole story, nor do we know how lignin is formed in the growing plant or how it is combined with other constituents of the vegetable structure. It is a definite responsibility of fundamental research to clarify the constitution of the lignin molecule and to tell us how it exists and how it is formed in plants. When we have this knowledge, we certainly should be able to apply it in the pulp and paper industry. Why is it that sulphur in some form is the only element capable of rendering the lignin present in plant life soluble and liberating the cellulose? It is true that certain organic liquids are capable of dissolving lignin under certain conditions, but these solvents are at present far too expensive as compared with suitable sulphur compounds. Much, however, remains to be done before all of our questions are answered.

Turning now to the subject of paper, it hardly seems necessary for me to point out the many problems which confront our industry and which can only be solved either by fundamental research or a practical engineering approach to these problems. Volumes have been written on the subject of beating, but in spite of this vast literature, there still remains much to be learned. How can we produce the best quality in a given paper with a minimum expenditure of power? How may added chemicals aid us in reducing the power cost per ton of paper produced? How may we improve the sizing of paper, keeping in mind that rosin and alum have for centuries been used for this purpose? New products and new methods are constantly being brought to our attention not only for the conventional sizing of paper but also for the production of high wet strength, so much demanded during the emergency of World War II.

I could probably go on for some time pointing out essential research in the pulp and paper industry. It is not my object to do so at this time. I only wish to emphasize the necessity of continued fundamental and applied research to keep our industry abreast of modern times wherein research is playing an ever increasing and important role.

The final address, "The Southern Pulp and Paper Industry and the Technologist," was given by Gunnar W. E. Nicholson, Vice-President of the Union Bag & Paper Corp., Savannah, Ga. Mr. Nicholson has been an enthusiastic supporter of the project of establishing a TAPPI section in the southeastern region. Mr. Nicholson's remarks follow:

The Southern Pulp and Paper Industry and the Technologist

Gunnar W. E. Nicholson

We are today gathered here to formalize the most recently established section of TAPPI, covering members of the national organization located in the three southeastern states—South Carolina, Georgia, and Florida.

It is pertinent to note that this section of the United States has the largest tonnage production of pulp and paper and the greatest concentration of pulp and paper technologists. As a matter of fact I believe I can truthfully say that this is probably today the most highly concentrated pulp and paper operation in the world. If the production of pulp mills now being built is included, the chemical pulp production in these three southern states, Florida, Georgia, and South Carolina, as represented by this section of TAPPI, amounts to approximately 5 million tons per year, or nearly one third of the total annual production of the United States. I estimate also that more than 600 technical graduates are employed in the pulp and paper industry in these three states.

It is also interesting to note that there was not one of the present pulp and paper mills in operation 25 years ago. Of course, as you know, our particular branch of pulp manu-

Gunnar W. E. Nicholson, Vice-President in Charge of Manufacturing, Union Bag & Paper Co., New York, N. Y.

facture by the kraft process was developed in the Old Country and brought over to this country as recently as 50 years ago, when the first kraft pulp mill was built on the American continent by a Norwegian engineer at East Angus, Quebec, Canada, in 1907, and it is still in operation.

It may have been just as well that this section of TAPPI was not organized earlier, as I am sure you now have a real solid foundation to build upon, and there is no reason why this section should not develop into the most active section of TAPPI in time to come.

Although we cannot expect a continuation of growth in tonnage in the future to the extent that we have in the past and present, there are all indications that we will see increased usage of all types of wood available in the southern states and also an increased efficiency in the usage of all species of wood for pulp manufacture. Estimates have been made that if slabs and trimmings from sawmills in the southern states could be used the yearly saving from this source alone would correspond to 5,000,000 cords of pulpwood. Indications are that we will see this being brought about in the very near future.

There are plenty of gumwood and other hardwoods available and these are being used more and more in the manufacture of semichemical pulp for corrugating media and similar grades, and we can predict that it is only a matter of a few years when pinewood will be used only for kraft pulp requiring long fiber and strength.

The greatest development in years to come will undoubtedly be in the quality of pulp, and here again hardwood will play a great role. I have many times made the statement that there is no grade of paper and board made today from chemical pulp which cannot be duplicated and made by the southern pulp and paper industry. We can today make two distinct and separate types of bleached pulp from gumwood and pine, and by mixing the two grades of pulp in various proportions before the stock is being refined it is possible to produce and duplicate any grade of paper made today from chemical pulp, and at a cost which cannot be duplicated anywhere else.

What I have said in regard to the growth and development of the pulp and paper industry in this section applies in the same manner to the other southern states. The growth has been tremendous, as approximately one half of the chemical pulp production in the United States is made today below the Mason Dixon Line, and the proportion is still growing.

The total domestic pulpwood production in the United States last year was 25 million cords, of which the southern states produced 56%, or 14 million cords. Considering the pulp mills now under construction located below the Mason Dixon Line, the pulpwood production in the southern states will undoubtedly reach two thirds or better of the total in our country. Of this the southeastern section—South Carolina, Georgia, and Florida—will produce about half.

It is also interesting to note that more than half of all lumber used in our country comes from southern operation.

I am quite sure you have read about the great potentialities of the forests in Alaska for the pulp and paper industry. It so happens that I visited Alaska and the Yukon during my vacation this last Summer. I saw a great deal of the beautiful Sitka spruce and hemlock forests growing in the temperate climate section from Prince Rupert up to Skagway. Puget Sound Pulp and Paper Co. jointly with the American Viscose Co. have just started construction of a pulp mill to make dissolving pulp at Ketchikan. The United States Forest Service is very optimistic about the future of the pulp and paper industry in that section, and they state that there is a perpetual supply of forest for the manufacture of 3000 tons of pulp per day. I am sure it is of interest to you to know that this is just about the production I expect to see in Savannah, Ga., alone when the present plants of the Union Bag & Paper Corp. and the Robert Gair Co. are completed. This brings

J. R. Lientz, C. E. Hartford, M. B. Barkley, R. T. Hudson, and Kirk Sutlive

out again the tremendous importance of the pulp and paper industry in this particular section.

Another figure which might be of interest to you is the import of pulp to the United States from European countries. The most recent figure is 125,000 tons during the first seven months of this year, or at an annual rate of approximately 200,000 tons. This figure before World War II was over 1,500,000 tons.

I do not feel we need be too concerned about shortage of raw material as the southern pulp and paper industry has taken the most prominent lead, particularly during the last ten years, in applying progressive and up-to-date forest management. This can probably best be illustrated by mentioning the fact that the yearly growth per acre has been more than doubled in the pulp and paper company-owned forests during the last ten to fifteen years, compared with the growth on the same type of land owned by other landowners in the same section. This undoubtedly explains the statistical fact that the growth per acre is today greatest in the southeastern section where we have the greatest concentration of pulp and paper manufacture. The pulp and paper industry is not only continuing and expanding this type of work on its own timber limits but is also giving a great deal of assistance to other landowners, particularly the small ones. I should like to state at this time that the various state authorities have been and are doing a great job in improving forests, particularly by providing good forest fire protection, and I am very pleased to state that the State of Georgia is taking the lead.

Our industry is today taking the lead in applying technical know-how in forestry management along the same lines as has been done during the last ten to fifteen years in the operation of pulp and paper mills. Forestry has become a permanent part of our economy. This probably can best be proved by stating the fact that more than $15 per acre of forest land has been so far invested in manufacturing enterprises for conversion of wood into finished products. The combination of advanced technology in the pulp and paper mills together with advanced timber culture in the woodlands is bringing about the most creative and dynamic forestry program in existence. There are no limits to the application of skill by the technologist in developing new processes and new products from wood.

We will in the future see a great number of engineers working together with trained foresters in our woodlands operations as well as forest management, particularly in forest research and development. We know very little today about the relationship of yield and quality of pulp to the growth of various species of pine, as well as various locations of growth. We are still talking about the number of cords of pulpwood required per ton of pulp and the number of cords grown per

R. G. Macdonald, T. T. Dunn, C. E. Youngchild, G. W. E. Nicholson, and J. M. MacBrayne

acre of forestland when we should be using terms of pounds of moisture-free wood per ton of pulp and tons of fiber grown per acre of forest land. There are so many unexplored territories waiting for scientific know-how to be applied that they are too numerous to be mentioned on an occasion like this. However, as an illustration I should like to mention a few:

What about the relationship between the density of wood and pulp yield and quality?

Why do we lose as much as 12 to 15% of the dry weight of wood in storage in the summertime? Just think of the millions of dollars wasted each year!

How can we improve growth of pulpwood by application of the know-how in genetics as has been done in increase and growth of farm products?

To what extent can improvement of growth by species or otherwise be improved upon by application of the know-how of soil chemistry?

I could continue like this for a long time.

Before I leave the subject of pulp and pulpwood I should like to emphasize that better utilization of wood from the forest as well as wood through the pulp mill are problems we must tackle in the near future, and I believe it would pay better returns to give more attention to such improvements than to try to get the very last out of the chemicals, power, steam, etc., in pulp conversion when we consider that the cost of pulpwood per ton of slush kraft pulp is more than half of the total cost. Looking upon such a splendid group of technical men as I do tonight, I cannot but think about all the interesting work you have before you.

I must not, however, overlook the reason for our get-together here tonight, and that is to celebrate the presentation of the charter making your organization a local section of the national organization of TAPPI. TAPPI is a very active organization with great opportunities for the members to participate in the various endeavors. As you know, there are more than 50 standing committees which provide the principal means within the organization for the collection and dissemination of technical knowledge. The service of the committee organization is entirely voluntary and provides opportunities to participate to any member who is willing to give freely of personal time to work. Active committee work will also invariably lead to participation in the association's various activities, and attendance at the various meetings is of very great help to every technical man in our industry, in order to keep abreast of the new developments, obtain a broader viewpoint, and enjoy the fine fellowship existing in the organization. It is also very helpful to young technical men in getting to know older men in more responsible positions. TAPPI is an organization with an outstandingly fine record, highly respected, and known all over the world. I know from experience that you can feel proud of membership in it, and I wish you and your local organization the very best for the future.

Pacific

The main objective of the visit of Dr. Karl Freudenberg to the United States was to give the lectures at the annual seminar sponsored by the Pacific Section. Incidental to this visit Dr. Freudenberg lectured at meetings of several TAPPI local sections.

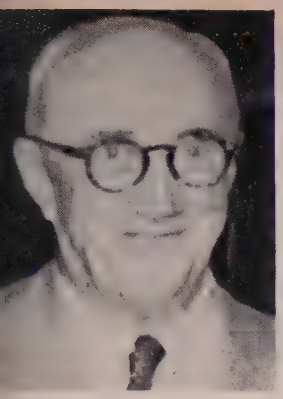
Dr. Freudenberg's lectures at the seminars held at Seattle, Wash., and Portland, Ore., follow in abstract:

Natural and Artificial Lignin

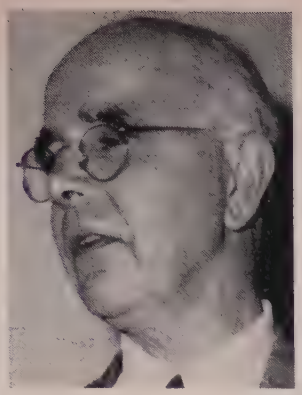
The relationship between conifer lignin and coniferyl alcohol has now been proved. Lignin, however, is not a simple polymerization product of the alcohol but a polymerization product of a coniferyl alcohol radical having one less hydrogen. Coniferyl alcohol can be converted into an artificial lignin by means of dehydrogenation enzymes. This process can be explained as a result of the fact that a coniferyl radical poorer in hydrogen is polymerized in aqueous solution automatically into water-insoluble lignin. The identity of this artificial polymer with natural lignin may be considered as being fairly well established. Such small differences as still exist may be explained as resulting from the aging processes of the lignin. It is possible to direct the transformation of coniferyl alcohol into lignin by a dehydrogenation enzyme in such a way as to produce a crystalline low-molecular intermediate which, on further action of the enzyme, is converted into lignin. Hitherto, a dehydrogenating enzyme of the edible mushroom, *Psalliota campestris*, has been used but more recently it has been possible to show that the enzymes of the tissues still in the state of lignification and of the cambium lying between the wood itself and the bark also convert coniferyl alcohol into lignin with the formation of the same intermediate. This intermediate is called dehydrodiconiferyl alcohol and its constitution has been established. In addition to benzene rings, it contains a heterocyclic ring with 4 carbon atoms and one oxygen (phenylcoumaran structure). It must be assumed that, in addition to the polymerization caused by a loss of hydrogen, there also occurs a regular polymerization in which other oxygen linkages are formed. These oxygen bonds combine the coniferyl groups through oxygen bridges (aliphatic ethers) as is shown by experimental results. This supports the concept of the Swedish school that the sulphite process consists, in part, of a reaction with aliphatic ether groups.

In order to explain the formation of wood it must be assumed that, in the few cell layers that are in the process of lignification and which are located between the cambium and

C. E. Tyler, G. E. Bosserdet, T. T. Dunn, and R. C. Sproul



C. E. Youngchild, who recently celebrated his 70th birthday



Karl Freudenberg, Heidelberg University

he finished wood, coniferyl alcohol is present and is converted nto lignin by dehydrogenation enzymes of these same cells. Because of its insolubility, this lignin remains in the cell walls nd converts them into wood. This raises the question of ow the coniferyl alcohol which is a very sensitive substance eaches the place of wood formation. It has long been known hat coniferin, the glucoside of coniferyl alcohol, occurs in the ambial sap. This glucoside is not attacked by dehydrogenaion enzymes. It was therefore necessary to look for another nzyme (glucosidase) which is capable of splitting coniferin nto its components, glucose and coniferyl alcohol. The folowing method was used for the detection of this glucosidase n microscopic wood sections: the precursor of indigo in the lant is the colorless glucoside, indican. Indican is cleaved y enzymes capable of splitting glucosides into glucose and olorless indoxyl which, in turn, is rapidly converted by the xygen of the air into the deeply colored indigo. The presnce of a glucoside-splitting enzyme in a plant section is roved if this section is stained blue when it is treated with a olorless indican solution. On application of this method to oung coniferous sprouts (spruce, pine, araucaria) it is found hat the indigo color appears in a very limited and strictly ocalized region within the cambial zone—in other words, in hat part of the tissue which is in the process of lignification ut not in the part already lignified. This forms the last link n the chain of proofs for the explanation of the lignification rocess. In the cambial sap formed somewhere in the plant is he glucoside coniferin. This diffuses into the newly formed ells and meets the localized glucosidase (glucoside-splitting nzyme), where the coniferin is split into glucose and coniferyl lcohol which, therefore, is liberated directly where the ligniication occurs. The dehydrogenation enzymes present then onvert the liberated coniferyl alcohol in this area into lignin.

Lignin in deciduous woods contains, in addition to the oniferyl component, sinapinyl alcohol, the glucoside of hich (syringin) has been found in deciduous woods—i.e., in he bark of lilac. The cleavage product of syringin (sinapinyl lcohol), when treated with dehydrogenation enzymes, gives lignin like product only in a very incomplete way. In nature lso no lignin is found consisting only of the sinapinyl comonent. However, when sinapinyl alcohol and coniferyl alcohol re mixed, a mixed polymer is formed, consisting of both comonents and identical in every respect with lignin from deiduous wood.

Organic Chemical Work on the Constitution of Cellulose

At the death of Emil Fischer in 1919, it was known that ellulose consists only of glucose units which are combined y a bifunctional reaction in a glucoside-like manner to form rge molecules. However, it was still unknown whether the ellobiose linkage is the sole binding principle and how large he chain molecules are.

In 1921 it was found that on hydrolysis about 60–70% of the glucose formed passes through the cellobiose state. This is in agreement with the assumption that all linkages are of the cellobiose form.

When the principle of optical superposition was found to be a distance principle it could be shown that this could be applied to cellulose and oligosaccharides which affords another proof of the uniformity of the linkages.

The synthesis of the crystalline methylated cellotriose occurs in the same way as that of methylheptamethyl-β-cellobioside, proving that the two linkages in the trisaccharide are the same as in cellobiose.

Since, on degradation of the methylated cellulose, only 2,3,6-trimethylglucose is formed, the following linkages are possible in addition to the cellobiose bonds: linkages of the maltose type, of the 5-(β-glucosido)-glucofuranose type, and of the 5-(α-glucoside) glucofuranose type.

From the measurement of the hydrolysis velocity of cellulose and its oligosaccharides and of maltose it could be predicted that, if maltose linkages were present, maltose should be found in addition to cellobiose on degradation of cellulose. This is not the case. The strong dextrorotation of the maltose bond would also make the application of the superposition rule impossible. 5-(β-glucosido)glucofuranose has recently been synthesized. Unlike cellobiose, it is strongly levorotatory and would be optically detected. 5-Glucosidoglucose linkages can occur in cellulose only if the same number of furanose linkages are present. This, however, is not the case because furanosides are hydrolyzed 1000 times more quickly than pyranosides. The cellobiose linkage is, therefore, the only type in cellulose.

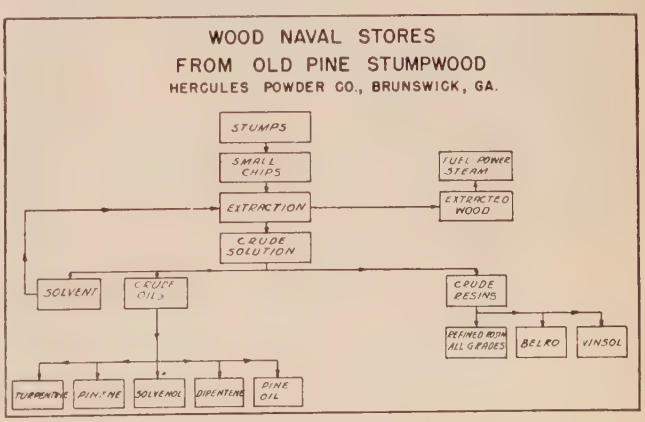
As for the elucidation of the chain length of cellulose by organic-chemical means, it can be stated that the chain is so large that, in a completely methylated cellulose, no tetramethylglucose can be found.

Delaware Valley

The Delaware Valley Section held its first meeting of the current season on Sept. 19, 1952. The meeting started out in the afternoon with a visit to the mill of the Container Corp. of America in Philadelphia, Pa., in which 75 members participated, and was followed by a panel discussion of "Cylinder Machine Operations." The evening meeting was held at the Engineers Club in Philadelphia.

Glen Renegar, Mill Manager of the Container Corp. of America, acted as moderator for the evening's panel with George Suydam, Superintendent of the Riegelsville Mill, Riegel Paper Corp; Thomas Brookover, Chemical Engineer, Downington Paper Co.; Harold N. Holcombe, Technical Control Engineer of the Schmidt and Ault Paper Co., as Panel members.

With Harold Brill presiding, the evening meeting was opened and members and guests welcomed to the first meeting of the current season. Chairman Brill expressed his appreciation for the courtesies extended by the Container Corp. of



America for having the Section as their guests for a visit through their mill during the afternoon.

At the meeting to be held on either December 5 or 12 Hugo H. Hanson, President, W. C. Hamilton and Sons, will deliver a talk on "Problems of Mill Management."

Jack Harper, Publicity Chairman, requested that Section members send him newsworthy items for possible inclusion in meeting notices. This is aimed at keeping the Section membership informed about other members and their activities. Cards suitable for sending this information were distributed. These were addressed to: J. C. Harper, in care of Dowingtown Manufacturing Co., Dowingtown, Pa.

John Weidner, former Section Chairman, had charge of many of the arrangements for this meeting. Harold Brill thanked him for his service and asked that he introduce Mr. Renegar, the moderator for the evening's panel.

Glen Renegar introduced the members of his panel, and he then explained that the choice of subtopics under the main subject, "Cylinder Machine Operations," was based on the responses to a questionnaire submitted to the membership to determine what their main interests were.

The first subject was that of "Cleaning Systems," particularly the use of Vortraps and similar equipment. Mr. Suydam stated that his mill employed Vortraps between the jordans and paper machines. He pointed out that proper installation and maintenance were most important to the successful operation of any equipment designed for the removal of foreign material from stock flowing to the paper machine. The use of diaphragm-type valves rather than gate valves; the control of pressure drop; and the prevention of entrained air with the stock in making dilutions were all important points to be considered. Mr. Suydam spoke of the use of Vortraps in series as being very effective, particularly in removing fine sand from rope stock, the sand being completely removed in the secondary section of the Vortrap system.

There is some wear noticed in the bottom cones of Vortraps in this application.

Mr. Brookover stated that considerable trouble was experienced with lower grades of paper stock as the dirt therein cuts out the seats. He pointed out that installation between the hydrapulper and the jordans gives better jordan filling life. The question of dilution, the stock furnished, and subsequent thickening for jordaning becomes an economic problem if such dilution is not part of the existing system.

Mr. Renegar stated that chrome plating of parts subject to heavy wear, such as the bottom cones, is quite effective in cutting down this excessive wear.

Mr. Holcombe spoke of experience in using automatic flap valves for cleaning where the operational characteristics varied due to wear. When newly installed, water losses were about 7 g.p.m.; whereas, when worn, this loss increased to about 60 g.p.m.

Question from the floor: "Should Vortraps be used for the liner or filler, or both?"

Mr. Brookover stated his experience had been only with the heavy and coarse foreign materials in mixed furnishes for board filler stock. Mr. Brookover continued by asking the panel: "What should an optimum dilution be; also, what would the capacity at the optimum points be in gallons per minute?"

Mr. Suydam believed it would be about 0.1% consistency but that this depended on the pump power involved; and that from 0.1 to 0.5% was probably the full effective range. 0.1% is recommended by the Nichols Engineering and Research Corp., one tenth per cent gives about 7 lb. of reject per ton of rope stock.

Mr. Brookover felt that this optimum point depends on the particular application and per cent consistency. He pointed out, however, that care must be exercised in any discussion so that economic optimum is not confused with removal efficiency. One and three tenths per cent consistency is said to be the satisfactory top limit in some board mill operations.

E. R. Smith, Union Mills Paper Manufacturing Co., uses Dirtecs. He originally started at 0.5 % consistency but found that 0.2 % was better and likely was the optimum.

Mr. Suydam operates both Dirtecs and Vortraps, but on different machines.

A. L. M. Bixler, Riegal Paper Corp., felt that 0.2 to 0.5 % consistency was about optimum considering all other factors.

Mr. Smith wanted to know whether the primary or the secondary removed the greater amount of dirt with a higher specific gravity.

Mr. Suydam believed that if the primary rejects a type of dirt, then the secondary would also remove it. However, the secondary stock is more dilute and should handle lighter dirt for that reason.

The question of cone life was raised.

Mr. Renegar's experience was that cone life was about two months with very heavy dirt and especially in stocks containing tramp material. For the barrel in the same application he would expect about a year.

J. D. Lyall, Armstrong Cork Co., asked: "What per cent of the original stock was rejected?" Reference was made to a paper on this subject appearing in *Tappi* magazine during 1951 in which the Nichols Engineering and Research Corp. pointed that at 0.5% consistency quite a bit of dirt was removed and that at 1.0 % there was good dirt removal.

Mr. Brookover brought up the question: "What is the ideal capacity per Vortrap?" He mentioned that Nichols recommends 250 g.p.m. for a 4-in. Vortrap.

Mr. Renegar stated that 1200 g.p.m. on a 10-in. Vortrap could be expected at 0.8 to 1.0% consistency.

Mr. Suydam stated that with greater pressure drops, such as increasing the drop from 25 to 35 p.s.i., there is better cleaning but also greater loss of good fiber.

The second topic brought up for discussion was "The Use of White Water in Machine Showers."

Mr. Holcombe stated that his company had tried city water to get better felt life but found that the cost and the disposal problems forced them to give this up. The other extreme was the use of straight white water from the vat, containing 2 to 3 lb. of suspended solids per 1000 gal. On checking the dissolved solids' effect in wash water, he found that removal of suspended solids and barring coagulation of dissolved solids the felt life was increased by 25%. Broughton oscillating needle showers were used. They found that water treated by a Hardinge filter with an automatic back wash contained essentially the same colloidal condition as the water in the cylinder molds.

Mr. Brookover brought up the problem of "How much effect does the temperature of the felt shower water have on the drying system when the felt is perhaps 80% soaking wet before the shower? The water flow is from the wet sheet to the felt and not vice versa. How much does this cool it off as compared to the cooling effect of the rate of evaporation?"

Mr. Holcombe stated that, disregarding cost, he felt that city water was to be preferred over treated white water.

Howard Wright, of S. Austin Bicking Co., pointed out that the use of an OCO water system definitely increased felt life. He further pointed out that felt manufacturers claimed that an acidified white water kept felts cleaner and more open especially after water was warm. There is a possibility of considerable savings both from the heat point of view and from the increased machine production that results. With steam at $1.00 per 1000 lb., an expected saving of from $25 to $50 per day is possible.

The question of foam was raised.

Mr. Renegar pointed out that in hard water areas the use of white water was better than fresh water when foam problems were encountered.

Mr. Wright asked the panel: "How much water was used in making a ton of board?"

Mr. Renegar answered that in the various folding box board mills around the country the figures varied from 6500

15,000 gal. per ton. He further stated that governmental agencies were generally requiring more closing up of white water systems; and were insisting that rejected water be cleaned up before being discharged into streams. The fact that in many regions the discharged water had to be of a better quality than that originally drawn from the streams was also mentioned.

Mr. Wright pointed out, however, that in some cases where board mills had been forced to treat the waste water, there had been actual savings made.

Mr. Brookover expressed the opinion that no heat savings could be claimed, or at least not full savings, if there were subsequent steam losses to the atmosphere. He further pointed out that efficient handling of the various processed water cycles is required to maintain economic balances in the more open systems.

John P. Weidner, Container Corp. of America, mentioned that governmental agencies have reported savings from 1 to 5% but that they have no interest nor do they report on the cost of making these savings. They report how much is spent on the outside but not how much is spent inside of reclaiming.

Mr. Renegar added that maintenance of reclaiming equipment is a big factor.

The third topic of discussion was "Testing Procedures."

Mr. Brookover commented that in his company they did not have many of the controls common to a large number of board mills. They have done some investigation on these controls and in one case were surprised to find that freeness did not change with consistency, and that sheet weight on the machine did not change to the same degree as the consistency at the distributor headbox. He further stated that they have obtained satisfactory results in sheet weight with stock varying in a range of $3^1/_2$ to $5^1/_2$%. At Downingtown the beatermen and machine tenders checked on pH control and sizing, as Dowingtown believes, that as much responsibility as possible for process control should rest with the operating crews.

Mr. Holcombe stated that at Schmidt and Ault consistency control for basis weight and refining had been attempted they found that there were so many variables in the stock system that consistency control was not the best answer in regulating basis weight. Fiber length, wire mesh of cylinder face, and hydration all affect operating characteristics.

Mr. Suydam mentioned that the stuff gate itself is a satisfactory controller; as consistency decreases, the volume increases tending to hold the weight.

The use of the classifier (Clark classifier manufactured by Thwing-Albert Instrument Co.) as a control on tensile was brought up from the floor. It was pointed out that this instrument could also be used to show the amount of degradation in bleaching.

Mr. Wright indicated that he knew of a newsprint mill that was at present using the classifier as a control. The ratio of optimum fiber length of groundwood required to obtain highest speed had been determined. The classifier therefore was used to measure this optimum fiber length. He pointed out, however, that this method was too slow for routine tests.

James Galloway, Riegel Paper Corp., stated that determinations were being made in 25 minutes after finding the range for one screen.

Mr. Brookover pointed out that in the case of blowing on the machine, freeness tests sometimes indicate the source of the trouble. However, many times difficulties in cases of blowing seem to be tied up with other unmeasured variables such as dilution, flow rates, etc. At Dowingtown, he indicated, they had done some classification work on mixed furnishes with no conclusive results.

John Weidner pointed out that from the discussion he would question whether research was being compared with

Delaware Valley Section group which visited the mill of the Container Corp. of America, Manayunk, Pa., on Sept. 19

routine control, after all the machine is the final test, but freeness should be uniform.

Mr. Renegar added that it is recognized that operating variables cause trouble; blowing and crushing can be due to consistency variations as well as freeness.

Mr. Suydam was asked what testing procedures were used in the beating of rope and hemp stocks. He pointed out there were never two shipments of raw stock alike. After cooking, broke stock was brushed slowly with a dull light tackle; this was later beaten and reduced in freeness from 720 to 100 ml. High-speed refiners are very poor for rope beating. To develop strength, it is better to brush slowly with a broad brush and gradually develop the strength.

Mr. Smith asked how sisal was beaten.

Mr. Suydam answered that sisal must always be beaten slowly. At Riegel cutting sisal fibers to equal $^3/_8$-in. lengths on a precision trimmer had been tried but this was found to be very unsatisfactory since no strength was obtained in the final sheet.

Mr. Wright asked if lava rolls were used for this purpose.

Mr. Suydam answered that he did not know of any.

Mr. Lyall, of Armstrong Cork Co., raised the question of the spacing of bars in beater rolls.

Mr. Suydam pointed out that in the early stages cluster bars were used. The stock is later treated with more bars. He pointed out that water hydration alone will give a freeness reduction, but this is not practical in production work.

Mr. Smith suggested smaller pockets on beater hoods, pointing out that with large pockets there are greater proportions of stock untreated.

The fourth topic of discussion was "What factors are important in vat design?"

Mr. Holcombe pointed out that not many vats that are installed keep their original design. The design depends on the grades run, the types of stock used, and the conditions imposed by the operation. Such factors as the vat circle, the velocity of approach, and speed of the machine have a big part to play in machine production. A vat designed for one weight and grade might be ideal but not for other weights and grades.

Frank Hollis had read a stimulating paper on vat design at a TAPPI meeting in New York. This is a good reference. There have been many changes but none have been universally accepted.

Mr. Renegar concurred and pointed out that vat design was a trial-and-error proposition. What may be good for one grade, may not be good for another, and that a vat system must be the result of compromises in design.

Mr. Phillip Goldsmith, Pusey & Jones Corp., stated that by controlling variables, the best possible formation on any machine can be produced. Bringing the stock to the wire uniformly without disturbing it will always give a quality sheet. Such a design will not be dead on the slowest stock nor too fast with the fastest stock. Consistency variation is the greatest factor. Pusey & Jones have a design of a double circle vat with the inner circle adjustable. This vat is designed for steam flow but may be used as counterflow as an expedient. The direct flow gives the best formation.

Mr. Brookover asked Mr. Goldsmith: "Do you mean that on the same cylinder on which machine making from 16 to 50 point board you would make the same number of tons per day? Is formation generally the bottle neck on speed?"

Mr. Goldsmith replied in the affirmative. He stated that by plotting optimum forming capacity against speed a means to translate speed to capacity may be developed.

Mr. Suydam asked how square a tensile ratio can be obtained on high speeds.

Mr. Goldsmith replied: "On a counterflow vat the tensile ratio is three or four to one. On a fourdrinier it is 1.5 to 1.0. On a direct flow vat it is 2.5 to 1.0."

Mr. Bixler asked "how can pipe lines be kept free and clean of slime and scale build-up?"

Mr. Renegar stated that at Container Corp. of America, that was accomplished with periodic wash-ups, use of chlorination, having vat lines pitched with no dead pockets. Piping being undersized or oversized resulted in bad conditions. The question of the type of furnish and how much raw water was being used, the type of cleaning of stock, and the materials of construction all played an important part in deciding how often wash-ups were required. Stainless steel is very good as the material of construction since it cuts down the resistance to flow among other things.

Mr. Wright pointed out that Lithcoted pipe was also a material which is kept clean easily and reduces pipe friction by an estimated 20%.

Mr. Renegar indicated that the use of pipe with too small a diameter is not what many people claimed from an economic point of view.

The last topic of discussion was "What can be done in the matter of waste paper classification?"

Mr. Suydam pointed out that wastepaper is a problem, but burlap is one of the worst waste materials handled today. It contains rubber, plastic, tar, and other objectionable materials.

Mr. Wright asked: "How can conformances to specifications be enforced?"

Mr. Holcombe replied that this can be affected by selecting suppliers and stabilizing the sources. However, one can't always be choosy. Wastepapers must be used when needed, and it isn't practical to store the inventory required to run a large mill. Consideration must be given to suppliers making it possible for them to have continuous outlets.

Mr. Brookover pointed out that quality control on wastepaper received at the rate of 200 tons per day is practically impossible. It is much easier to police wire grades. At Downingtown each bale is tagged and a record of its use is kept. All machine samples of board are examined thoroughly. This has enabled them to find the culprit and take action against repetition. This procedure, however, must be continuous to be effective.

Lake Erie

The Lake Erie Section met on September 19 for the first meeting of the year at the Hotel Carter in Cleveland, Ohio. This is a new meeting place for the Section and it will provide improved facilities for our meetings.

The subject for the evening, "They're Always Judging Me," was given by E. L. Stromberg, Chairman, Dept. of Psychology, Western Reserve University. Dr. Stromberg's background in this type of work is Lt. Commander, U.S.N., Aviation Psychology, Assistant Professor of Psychology, Oklahoma A & M, and Industrial Psychologist, Stevenson, Jordan, and Harrison, Management Consultants. Dr. Stromberg is a forceful speaker and his inspiring talk left everyone with the resolution that he would improve his relationships with his fellow employees and subordinates.

Dr. Stromberg pointed out that there isn't a company in the country which doesn't take great care in the selection of any equipment it is going to purchase or the materials they are going to use in their manufacturing process. However, when they select their employees, it is often another matter. Too often they are chosen because of their outward or physical appearance, such as, how does he comb his hair, how does he walk, does he sit up? How often is it that they are chosen on how well qualified they are to do a job?

Supervisors are always ready to judge the people working for them but do they realize that they are being judged too and are they willing to accept the judgment that is given them? Surveys have shown that two things employees consider most important in their bosses are how much does the boss know about the job he is handling and how considerate is he of people working for him? Training programs are often used these days to improve the employee relationships. It is often asked, how effective are these training programs and are they accepted? The programs are very worth while but be sure the instruction is started at the right end of supervision. Foremen reflect the attitudes of their supervisors. Thus, if a man goes to school and learns new methods and, when he returns, tries to put them to practice in an organization which doesn't practice those principles, he doesn't have a chance. Before long, he will be back to the methods that the rest of the people in the organization use. Be sure the program is started at the top. Where the boss accepts the principles taught in these training courses, the men below him will follow.

When looking for a man for a job, try to find out how the prospect thinks, how he reacts to various situations, does he have ideas of his own, does he stick to a task until it is completed, will he stand up for his principles? Don't just say he looks like a good man.

The discussion period was very enthusiastic and brought out considerable discussion on specific cases which had come up and how they were handled.

L. E. Rolig, *Secretary*

Ohio

Dr. Karl Freudenberg of Heidelberg University, Heidelberg, Germany, talked to two very interested groups of TAPPI members at the Manchester Hotel in Middletown, Ohio, on Monday, Sept. 15, 1952.

In the afternoon he conducted a round table for a group of 30 of the more serious organic chemists on the subject "Research on Natural and Artificial Lignin and Its Constitution." In the evening he spoke before a group of 96 individuals giving a talk in less technical terms on "The Formation of Lignin in Plant Tissues."

The magazine *Ohio Tappi* now has a staff of reporters to collect news items consisting of:

M. G. Lyon, Champion Paper & Fibre Co., Co-editor
D. F. Jenkins, Champion Paper & Fibre Co., Co-editor
John M. Fisher, Mead Corp., Chillicothe, Ohio
Paul Schriber, 300 Wonderly Rd., Dayton, Ohio
Carl Gehring, Staley Starch Co.
Ernest Barker, Oxford Miami Paper Co., Historian

This magazine is issued each month to give the meeting date and information about the speaker, news of members and dates of future meetings.

The Nov. 18, 1952, meeting of the Ohio Section will be held at 6:45 p.m. in the American Legion Building in Middletown

Ohio. It will be a joint meeting with the Superintendents Association. The speaker will be H. B. Thacker of the Industrial Engineering Dept. of The Westinghouse Electric Corp., East Pittsburgh, Pa. His subject will be Electric Distribution Systems."

GEORGE. B. GREGG, *Recording Secretary*

Lake States

The Lake States Section held its first meeting of the 1952–53 season on September 10, at the Conway Hotel in Appleton, Wis., with a total attendance of 162, representing a good cross section of the Section membership.

Following the dinner, Chairman H. W. Rowe introduced the Section officers: Vice-Chairman, R. J. Seidl; Secretary, L. A. Moss; Treasurer, S. R. Parsons, and Executive Committeemen A. P. Adrian, G. K. Dickerman, K. W. Fries, Donald MacLaurin, and Reinhold Vogt.

Plans for the year's programs were then announced as follows:

Nov. 11, 1952—Institute of Paper Chemistry, Appleton, Wis., as host and Donald MacLaurin making arrangements. Guided tour through the Institute. Panel discussion on "Papermaking Properties of Fibers." Dinner at Conway Hotel at 6:30 p.m. with after dinner speaker on the subject, "Americanism."

Jan. 13, 1953—Conway Hotel, Appleton, Wis., at 6:30 p.m., dinner and talk on "Stainless Steel in the Paper Industry."

March 10, 1953—Green Bay, Wis.—Evening meeting on "Savealls."

April 15, 1953—Wisconsin Rapids, Wis., Nekoosa-Edwards Paper Co. as host with Chairman H. W. Rowe making the arrangements. Afternoon and evening meeting with visit to Nekoosa-Edwards Paper Co. followed by technical session at Elks Club, Wisconsin Rapids, on "Fourdrinier Operation and Defects Attributable to Same," dinner and after-dinner speaker.

May 12, 1953—Annual social meeting. Place and program to be announced.

Fritz Brauns of The Institute of Paper Chemistry then was called upon to introduce his long-time close friend and co-worker in the field of lignin and cellulose chemistry, Dr. Karl Freudenberg, as speaker of the evening.

Dr. Freudenberg is Director of Chemisches Institut, University of Heidelberg, Germany, and a well-known world authority on the structure and chemistry of lignin, cellulose, and tannins. The Lake States Section felt most fortunate to be able to arrange for such an outstanding speaker for its first meeting of the season.

Dr. Freudenberg spoke on the subject of "Lignin and Its Formation in Plant Tissue," using photomicrograph slide illustrations. His scholarly discussion included the latest theories on the structural formula of lignin. The formal speech was followed by a question-and-answer session in which many members of the audience participated.

L. A. MOSS, *Secretary*

Chicago

More cooperation between the paper manufacturer and the consumer was stressed by O. H. ("Doc") Runyan as he addressed the September 15 meeting of the Chicago Section. Mr. Runyan, field secretary for the Graphic Arts Association of Illinois, Inc., spoke on the topic, "Paper at Work."

Suggesting that joint meetings between papermakers and the ultimate user (i.e., pressmen) would be of invaluable benefit to both, the speaker declared that the printer and the mill man should know more about each other. He said that whereas to the mill man paper represents the finished product, to the user it is nothing more than the raw material. The printing industry as a whole, he continued, is lacking in the knowledge of paper selection and the methods of making it behave. In this respect, Mr. Runyan added, the mill man doesn't know much about the end requirement.

Mr. Runyan, whose talk inaugurated the Section's 1952–53 season, described the paper selection course sponsored by the Graphic Arts Association. As director of this education project, he pointed out that he has seen the great benefit that such a program has to offer in acquainting all phases of the field—from mill to consumer—with each other's needs and problems. The course covers the subject in its entirety, from technical discussions of manufacturing processes to trade terms and proper mill-buyer relationships. Typical examples of difficulties arising between buyer and seller as the result of avoidable misunderstandings were described by Mr. Runyan, who has had 40 years' experience in the paper and printing industries.

Briefly describing the paper selection course, the speaker listed the various topics covered in the educational program. These include the physical characteristics of paper; a review of trade customs and purchasing; methods of testing; the relation of paper to printing plate; gravure, offset and letterpresses; the bindery; differences in pulps and their relation to the end use, and printing qualities.

Vance V. Vallandigham, chairman of the section, was in charge of the meeting. Mr. Vallandigham, Technical Director of the paper department of Kelco Co., was recently appointed Manager of Research and Development for the paper division of Potlatch Forests, Inc., Lewiston, Idaho.

D. W. ZIEGLER, *Publicity Committee*

LETTERS TO THE EDITOR

Corrugated Board Containers

To the Editor, *Tappi:*

We are enclosing herewith a memorandum on information required in respect to and data pertaining to the design of corrugated board containers for canned and preserved foods. We should be appreciative if you could assist us with advice and answers to the questions contained therein.

The problem comprises: (a) the design of a corrugated board of optimum strength involving the minimum weight of paper, and (b) the structural design of the carton.

(a) To obtain the maximum strength out of a corrugated board it is essential that the quality of the paper going into its construction be of the highest. It has been the custom of the industry in this country to prefer kraft liners and kraft fluting but, on occasion, other papers have been used, including Chemfibre and Italian straw paper. It is proposed that a specification be laid down for the paper so that if any substitute for kraft has to be used then its qualities should be at least equal to those of kraft.

Information is sought on the relative merits and details regarding kraft cylinder paper and fourdrinier kraft, as compared with ordinary kraft.

Information is also sought as to whether there are any modern substitutes for silicate adhesive in building up a better corrugated board.

A schedule is hereto attached, showing the gross weights when full of the more common cartons at present being used in the industry.

It has been the practice for cartons designed to go overseas to specify a 145-lb. grade of board for three of these cartons, viz. 24/A.2, 48/1-lb. Jam and the 48/A.1 (11-oz.)

All the others have been specified as 167-lb. board. The weights, of course, refer to the total weight of the paper (only) used in making the board, and in respect of 1000 sq. ft. of finished board. Calculations are based on the following table:

Kraft (including Chemfibre), lb. per 1000 sq. ft.	*Equivalent caliper*
26 = 1000 sq. ft.	0.009
33 = 1000 sq. ft.	0.012
42 = 1000 sq. ft.	0.016 Dry finish
47 = 1000 sq. ft.	0.016 Wet finish
64 = 1000 sq. ft.	0.020
69 = 1000 sq. ft.	0.023
90 = 1000 sq. ft.	0.030

plus an assumption that the fluting causes an additional 50% of paper per 1000 sq. ft. to be used. Almost exclusively the fluting has been 0.009 caliper, i.e., 26 lb. per 1000 sq. ft. in the flat, but 39 lb. after fluting. Thus a 145 board would be composed of:

Caliper		*Lb.*
An 0.020 kraft	=	64
An 0.009 kraft fluting	=	39
An 0.016 dry finish liner	=	42
		145

and a 167 board would be composed of:

An 0.020 kraft liner	=	64
An 0.009 kraft fluting	=	39
An 0.020 kraft liner	=	64
		167

The relationship of these specifications to the bursting strength either by Mullen or Cady test has not been clearly correlated or specified. Information is sought on the relationship, if any, between the various bursting strengths of materials in pounds per square inch, measured by the Cady or Mullen test, and the weight specification per 1000 sq. ft. or caliper specification. Recommendations as to routine instruments which should be made available to the factory laboratory are sought. Advice is also sought on simple equipment for making the following tests:

(a) Material and board values:
- (I) flat crushed test (corrugated flutes)
- (II) bursting strength—Mullen or Cady
- (III) G.E. (Beach) puncture and stiffness
- (IV) Elmendorf tear test
- (V) weight per sq. ft.
- (VI) caliper or thickness

(b) Box values:
- (I) compression—resistance to stacking loads or thrust cars etc.
- (II) rough handling
  - (a) revolving drum
  - (b) drop
  - (c) conbur incline impact
  - (d) vibration

Note: It is realized that equipment for testing under B(II) must be fairly extensive, and the question is "Is there any alternative approximate method of arriving at an indication of how the cartons will stand up to rough handling without extensive equipment?"

It has been the practice also, for canned and preserved foods, to use an A-flute, i.e., 36 corrugations to the linear foot but, while apparently the standard depth of such a corrugation should be $^3/_{16}$ of its height, there has been no standardization either of the contour of the flute or of its exact height. No information is available as to whether a B-flute, i.e., 51 corrugations to the linear foot, would not be better for the purpose, and guidance on this particular aspect is sought.

Confirmation is also sought on certain ideas which have suggested themselves and which are set out below:

It is considered that the effective strength of the board can be greatly improved by utilizing a heavier fluting at the expense of the inner liner. Thus a board of the following specifications has been made up and found to be very promising:

An outer liner of 0.023 caliper	=	69
A fluting of 0.016 dry finish	=	63
An inner liner of 0.009 caliper	=	26
Giving a total of		158

This appears to be more resistant to the strains found in our industry than the 167-lb. board, and comment is invited on its construction. It is based on improving resistance to the cans "digging in" into the board and is also based on the as yet unproved supposition that the cans will not go through a 0.009 caliper inner liner. Experience in packaging millions of cartons of the original specification would tend to indicate that this is most unlikely, but expert advice is sought, particularly as to the specification of a 0.009 liner which would stand up best to the conditions described.

(b) Structural design of the carton: The attached schedule shows the commonly accepted weight specification of board in relation to the gross contents, and criticism is invited. The problem is seen purely from the local canners' point of view, in that the carton is expected to fill several functions, some of which mitigate against each other.

It is common practice in this country to have the cans brought from the can maker to the canner in the cartons. In order to accommodate a slightly increased width, due to flanges, the cartons are made slightly oversize. They also suffer by virtue of the flaps being interleaved, and criticism here would be appreciated.

Also comment is invited on the method of bottom-stitching cartons, as against gluing.

Because of the slightly excessive dimensions, and because of the cans "digging in" into the board on stacking, a tight pack is almost impossible to achieve. It becomes necessary to minimize the adverse factors and, because millions of cartons have been successfully handled and shipped under what are admittedly disadvantageous conditions, we are led to the conclusion that improvement along certain lines will give a really satisfactory pack.

Failure of cartons has been caused mainly by tearing down the shortest side seam. While this is obviously the weakest part of the carton, the shape of the carton in most cases cannot be altered but must be preferably a two-layer arrangement, because of automatic filling equipment. This tends to give a shallow carton in many cases which, though ideal for stacking, gives a comparatively short side seam and therefore aggravates the weak portion. It is practice to have the corrugations running vertically up and down the stacked cartons, instead of horizontally. It is thought that by having the corrugations horizontally, tearing along the weak seam will be greatly minimized but that the compression stability of the box will be affected. Advice is sought here as to the best solution of the problem because, admittedly, the cans inside the carton support the carton and the corrugations are not called upon to withstand high stacking on their own. Because of the relatively loose pack, plus "digging in," the cartons "belly" to a certain extent, which would be exaggerated if the corrugations ran horizontally but which takes place in any case on storage, even with the corrugations running vertically. It is thought that the advantages of horizontal corrugations outweigh the disadvantages, and an authoritative opinion would be greatly appreciated. To support this viewpoint, the strengthening of the fluting is considered, either by using a 0.016 flute or by adopting a B-flute corrugation, i.e., 51 corrugations to the linear foot, each of the corrugations being $^1/_8$ in. high. It is considered that resistance to "digging in" is more important than cushioning the effect and that the B-flute might, therefore, be more satisfactory. Advice on the possibilities or desirability of either of the two suggestions to strenghen the fluting, by expert authority, is sought.

Guidance on the creasing practices both in the manufacture of the carton and in its subsequent handling, is invited. It is appreciated that offset scores are necessary, and that alteration to the structure of the corrugated board must bear in mind the danger of putting strains on one or other of the liners. It is thought that a properly creased board with a 0.023-0.016-0.009 caliper specification, will give no trouble at all, in spite of the heavier paper. Comment is invited on

Schedule of Carton Details

	Gross weights, lb.	*Inside dimensions, in.*	*Weight of board, lb.*
24/A.2	37	$13^5/_8 \times 10^1/_4 \times 9$	145
24/A.$2^1/_2$	55	$16^1/_8 \times 12^1/_{16} \times 9^3/_8$	167
48/A.1 tall	62	$18^1/_8 \times 12^1/_8 \times 9^3/_8$	167
6/A.10	48	$18^1/_2 \times 12^1/_4 \times 7^1/_8$	167
48/A.1 (11-oz.)	43	$16^3/_{16} \times 10^{13}/_{16} \times 8$	145
24/2-lb. (jams)	57	$16^1/_8 \times 12^1/_{16} \times 8^1/_4$	167
48/1-lb. (jams)	58	$12^1/_4 \times 12^1/_4 \times 10^3/_4$, "all flaps to meet"	145
6/8-lb. (jams)	55	$18^1/_2 \times 12^1/_4 \times 7^1/_8$	167

this particular specification and in relation to optimum specifications which might be suggested.

We would welcome an expression of opinion on the desirability or otherwise of metal strapping corrugated fiberboard containers containing canned products. Some canners use a round metal wire, others a flat strap, others use nothing—relying on proper gluing of the flaps to secure the package. The disadvantages of metal strapping are that there is a tendency to become slack either because the straps are used to lift the cartons by workmen handling the cartons in transit, or, alternatively, if the straps are pulled too tight they cut into the board and so weaken it. It would be desirable to know what practice is adopted in the United States.

B. Gershill, *Manager*
Standard Canners & Packers Ltd.
Port Elizabeth, South Africa

Editors Note: The following letters are responses from individuals who offer suggestions to Mr. Gershill. For the purpose of publication their identities are not revealed.

To the Editor, *Tappi:*

This is in reply to the letter dated June 4, from Standard Canners & Packers, Ltd., South Africa.

Unfortunately, we do not make any can cases here and so I am not completely up to date concerning these products. However, I have had some experience in the past which may be useful to them. The specifications shown show higher weight liners than are usually used in this country. I think 42 or 48-lb. liners are most generally used for can cases in this country. The suggestion of making unbalanced cases is logical from the technical standpoint although we found that it was very hard to avoid a severe curling of the board with unbalanced liners. I believe that the same would apply to the build-up of the corrugating material where the two outer liners are unbalanced although I have never seen tests to substantiate that belief.

We have made many tests on A against B-flute for can cases and were never able to show any appreciable difference. However, we did find that vertical grain corrugated can cases gave much better results than horizontal grain can cases. This means that the corrugations will run horizontally instead of vertically. For most can cases we feel that compression strength is not necessary and, therefore, find that the B-flute horizontal corrugation box with balanced liners is the best combination both from a technical and manufacturing standpoint.

In this country many combiners are equipped with a right angle delivery so that the blanks come off the corrugated combiner and go directly into a slitter so that they can be creased in a continuous operation rather than having to go to a separate slitter because of the horizontal corrugation. Of course, if glass jars or something requiring compression resistance is being packed then an A-flute vertical corrugation case is to be desired.

As far as tests are concerned we believe that the most important test for can cases is the tensile test across the score line. The final test in this case is the revolving drum but once tests have been completed in a revolving drum then it is possible to establish specifications for the liners and the completed corrugated board which can be made with the smaller laboratory instruments such as Mullen test, tensile tester, scales for determining weight per thousand square feet, and calipers.

It is also common practice in the United States to have the cans brought from the can maker to the canner in the cartons. We, therefore, have much the same problem that you have.

Our tests have never shown any great difference between stitching and gluing the bottom flaps. I am attaching a list showing the source and approximate prices of some of the testing equipment which we use and which might be valuable to these people. The prices shown are not necessarily up to date. The conbur impact test for shipping containers is normally constructed by the user and is covered in TAPPI Suggested Method T 801 sm-44.

To the Editor, *Tappi:*

Mr. Gershill has a list covering the field. I'll hit only basic principles.

The U. S. corrugated industry has developed cylinder and fourdrinier materials of comparable performance characteristics. Identity by the terminology used in his letter would restrict usage of materials that would equal or surpass the grades listed.

Starch adhesive is more widely used than silicate at the present time.

Weight of board has been discontinued as a common specification since certain combinations perform better than others with difference weights of materials. Performance usually determines what combinations are used commercially.

Rule 41 of the Railroads specifies the bursting strength of board by weight of commodity and size of container. Either the Mullen or Cady tester is recognized. Mullen testers are most commonly used in the industry.

The flat crush tester most commonly used is the Hinde & Dauch flat crush tester.

The Cady micrometer is the most commonly used tester for caliper.

Test data relationships for compression, conbur, etc., are all determined for specific hazards of shipment, and there is no simple formula.

Most can cases used in the states are usually B or C flute which is contrary to his statement.

It is suggested that commercial can cases be obtained by his firm to answer his material strength problem.

He has covered many operating details that suppliers such as ourselves cannot divulge. It would seem advisable for him to contact the National Canners Association or a commercial testing laboratory.

To the Editor, *Tappi:*

In paragraph (a) of Mr. Gershill's memorandum, he seeks information on relative merits and details regarding kraft cylinder paper and fourdrinier kraft as compared with ordinary kraft. Compared to the terminology used in this country, this is a little ambiguous, although it is my understanding that what they term "ordinary kraft" is similar to the usual filled kraft or jute sheets of this country. We consider kraft cylinder paper as being 100% virgin kraft, to be made on a cylinder type machine, as compared to the fourdrinier kraft which is also 100% virgin kraft and made on a fourdrinier machine. The main differences between these two sheets are the effective grain direction on the cylinder sheet, giving you a lower tear in the machine direction, a higher tear in the cross direction, generally a slightly lower Mullen, a higher end-to-end compression strength when fabricated into corrugated boxes, a lower top-to-bottom compression, and lower score

strength along flap scores. If my guess is right, that which they term "ordinary kraft" is our jute. Then much of the same applies on a weight-for-weight basis. Each of these strengths will be lower than the cylinder or fourdrinier, depending on how the "kraft" sheet was made.

Substitute for silicate: There has been considerable work done with a varied amount of results on the adding of clay to silicate to approved stiffness or building up the corrugated board. In many plants in the United States, silicate adhesive is not in use and has not been for some time. It has been replaced by starch which for the most part has been applied according to the Stein-Hall process. This process consists substantially of suspending raw starch in a partially cooked mixture, starting the bond with a wet starch and depending upon the heat of the hot plates and single facer to cook the raw starch, absorb the water present, and thereby create a very fast, strong bond. Urea-formaldehyde resins have been added to the starch as well as some of the resorcinol type. These resins give a waterproof bond which is desirable when the board is to be used for export shipping or for cold storage.

On page 2 of his memorandum, he requested information on the availability of simple equipment for making tests:

1. Flat crush test: Testing Machines, Inc., manufacture a machine developed by Hinde & Dauch which has the limit of 350. There is a heavier compression machine made by National Forge & Ordnance as well as a larger one made by Tinius Olsen. The Hinde & Dauch machine is the simplest of these three but is limited for capacity, and since most B-flute is run over 350 lb. on a 10-sq. in. sample, this machine is not too flexible.

2. Bursting strength: Either the jumbo Mullen or a Cady machine is a good instrument and comparable results can be obtained.

3. G.E. Beach puncture: There is only one of these machines made, namely the Beach machine.

4. Elmendorf tear test: The weighted pendulum made by Thwing is advisable, but on some linerboard, particularly cylinder sheets, it is difficult to get satisfactory results.

5. Weight per square foot: There are several units available. One of the simplest is made by Toledo Scale. A more precise instrument is made by Thwing. This machine must be mounted in a fixed position on a stable wall as the bearings are jeweled bearings and very sensitive.

6. Caliper or thickness: Either a Cady or a Schopper.

7. Compression: A compression machine is made by Tinius Olsen and also National Forge & Ordnance. On rough handling, the revolving drums are frequently made up by the firms themselves according to the specifications drawn up on ASTM of TAPPI.

8. Drop: I believe the machine is made by Acme Steel Co.

9. Conbur: These are made up by the firm desiring test.

10. Vibrator: Manufactured by L.A.B. Corp., Summit, N. J.

All of these last four machines are quire expensive and demand a lot of space. It has been proved that for can boxes especially, a very good indication can be arrived at by using a score strength test on a Mullen. This test has been developed by and is in use commercially by Don L. Quinn Laboratories, Chicago, and the exact procedure can be obtained from them as well as their comments on the advisability of using testing in preference to other methods of rough handling tests.

It is interesting to note that A-flute is in use in Africa for can boxes, whereas in this country B-flute is much more common. This is desirable, since the B-flute will give one a higher end-to-end compression than A-flute. A-flute will give one a top-to-bottom compression; but in can boxes, since the cans carry the vertical load, top-to-bottom compression is not needed, but end-to-end is needed to absorb rough handling in the end-to-end manner.

It is also interesting to note that you are recommending a sheet made of 0.023 liner on the outside, a fluting of 0.016 and an inner liner of 0.009. It is the general consensus that such an unbalanced sheet would be very hard to run in the manner in which we run corrugated board. Such a sheet manufactured at 400–450 f.p.m. would end up like a bunch of barrel staves. It is also probable that we would have trouble meeting Mullen test according to Rule 41, since the Mullen in the "in to out" will be low; and unless you have a very good sheet of 0.009 and the fluting is not damaged at all during corrugation, these tests will be considerably less than 200; and if more than one out of six bursts are below 200, the board is not considered acceptable. It is also my opinion that you would have considerable damage along the flap score—much more so than you would have with the 167-lb. board which is balanced.

In the structural design of the carton, it is quite a common procedure in this country to seal the bottom of the carton at the can manufacturer's plant, leaving the top closed but unsealed. I do not remember seeing any of the can plants shipping cans with the flaps interleaved, as there is bound to be damage to the flap scores in so doing. As I mentioned above, the can manufacturer bottom-seals the cases, they are inspected before being placed in the car, the boxes are closed but not sealed, and then placed upside down for shipment. In certain plants where bottom sealers are not available and it has been deemed necessary to seal the bottom of the case before placing the contents into it, bottom stitching has been used. But on can boxes, the bulk of the shipments are made with both top and bottom glued. In most cases, corrugations are vertical of the cartons, although there has been some work done on can boxes with horizontal corrugations and utilizing an opening, but so far this has never taken hold. Theoretically, it should be advisable to have horizontal corrugations and even an end opening, since you would experience an economy in the amount of board used, and as you mentioned, you do not need the top-to-bottom compression.

As I also mentioned earlier, B-flute is used by most canners rather than A, because not only do we get a better end-to-end compression, but the amount the board will compress between cases is reduced, and consequently one will experience a smaller change on the inside dimensions than with A-flute. In regard to offset scores, they are used on double wall boxes and on high test, as 350 and up, but are not generally used on the ordinary can box.

In regard to strapping, there is virtually no strapping done in this country on can boxes as this is not deemed necessary and will only entail additional expense on items which are considered small margin items.

RECENT BOOKS

Copolymerization. By Turner Alfrey, Jr., Dow Chemical Co., John J. Bohrer, International Resistance Co., and H. Mark, Polytechnic Institute of Brooklyn. Interscience Publishers, New York, 1952. Cloth, 6 × 9, 269 pages. $6.80.

This book is Volume VIII of the High Polymer Series of Interscience Publishers. It is the first systematic account of all copolymerization investigations of the experts who were closely connected with the development of this branch. It is by no means an elementary treatise; it is rather an excellent record of the progress that has been made in the growth of knowledge concerning synthetic resins. It features major contributions by the three authors including the reactions at the carbon-carbon double bond, kinetics of copolymerization, monomer distribution. The aim o

the book is to arrive at rules for the tailoring of copolymers of a desired structure and for establishing a relationship between the proportion of the monomers and the steering of copolymerization process to the properties of the final products.

Chemistry of Synthetic Dyes. Vol. I. By K. Venkataraman, Dept of Chemical Technology, The University Bombay, India. Academic Press, Inc., New York, 1952. Cloth, 6 × 9, 704 pages. $14.50. (Vol. II is in preparation.)

Dr. Venkataraman's book is an up-to-date monograph on a subject that has not heretofore been covered. In addition to the dealing with the chemistry of intermediates and dyes, it also covers the application of dyes, the relation between color and chemical constitution, the action of light on dyes, and the chemical constitution of dyes in relation to their affinity to fibers. Although the book deals to a considerable extent with the textile industry it is also useful to papermakers.

Chemical Engineering Operations, By Frank Rumford. Chemical Publishing Co., New York, 1952. Cloth, 5½' × 8½, 376 pages. $7.50.

This book, which uses European terminology, deals with the operations of chemical plants. Considerable attention is given to the practical aspects of plant design and operation. Although such subjects as fluid flow and power generation are omitted, it gives good coverage to such subjects as pumping, heat transfer, distillation, gas absorption, extraction, evaporation, drying, crystallization, filtration, centrifuging, size reduction, screening, and automatic control. 200 illustrations are shown.

Engineers' Illustrated Thesaurus. By Herbert Herkimer. Chemical Publishing Co., New York, 1952. Cloth, 5½ × 8, 572 pages. $6.00.

This is a remarkably useful book, containing as it does, over 8000 illustrations of machine elements and assembled machinery for the engineer, designer, draftsman, and manufacturer. Every conceivable type of machine element is clearly produced and identified. Every young engineer should compile a book of data sheets that may be found useful later on. It is a well-known fact that most complicated mechanisms consist merely of combinations of the six fundamental machines; pulley, wheel and axle, inclined plane, wedge, screw, and lever, yet the field of mechanisms and structures seems almost unlimited. The main sections cover fasteners, adjusting devices, structures, basic mechanical movements, derricks, conveyors, combustion, prime movers, electric appliances, and air conditioning.

Soluble Silicates. Vol. I. By James G. Vail, Director, The Philadelphia Quartz Co., New York. Reinhold Publishing Co., 1952. Cloth, 6 × 9, 357 pages. $9.00.

"Soluble Silicates" is American Chemical Society Monograph No. 116 and succeeds a previous book on the subject by the same author in 1928. Because of the large number of references it became necessary to issue the present work in two volumes. Volume II is in preparation. The first chapter deals with the history of the subject back to 1621, on commercial forms of silicates. There are 29 liquid silicates regularly made. An important item of this chapter is a discussion of methods of analysis. Very little information of this kind appears in the literature. The remaining chapter deals with homogeneous and heterogeneous systems and complex silicates systems. The preparation of the book required three years of concentrated effort by the author.

Pathology in Forest Practices. 2nd Ed. By Dow Vawter Baxter, Univ. of Michigan. John Wiley & Sons, New York, 1952. Cloth 6 × 9, 601 pages. $7.50.

Since tree diseases play such a vital role in tree production and utilization, their prevention and control is of great importance to the forester and others. The origin, nature, and causes of diseases in forests in the United States and Canada are discussed. It shows how to check, regulate, and control pests and diseases that limit the usefulness of trees. Considerable attention is given to fungi, forest nurseries, forest plantations, site, and cultural practice in forestry, decay, and discoloration.

Forestry and Its Career Opportunities. By Hardy L. Shirley, Dean and Professor of Forestry, College of Forestry, State University of New York, New York. McGraw-Hill Book Co., 1952. Cloth, 6 × 9, 492 pages. $6.50.

Dean Shirley has prepared this book for the benefit of laymen and students who may wish to follow forestry as a life career. It is one of the volumes in the American Forestry Series. It covers such subjects as: forest regions, forest care, harvesting timber crops, wood nature and uses, wildlife, protecting coil and watersheds, employers of foresters, education, trends, etc. One chapter is devoted to wood chemistry, papers, and plastics which briefly discusses the manufacture of paper and plastics. Mention is made of the work of TAPPI in relating plastics to paper.

Encyclopedia of Surface Active Agents. By J. P. Sisley (translated from French by P. J. Wood). Chemical Publishing Co., New York, 1952. Cloth, 6 × 9, 540 pages. $15.

Handbooks of this type are very useful for reference purposes. Part I deals with the general aspects of surface-active agents, their properties, applications, and methods of manufacture. Among the chemicals covered are wetting agents, detergents, penetrants, foaming compounds, emulsifiers, and dispersing agents. Part II is an alphabetical listing of brand-name surface-active agents manufactured throughout the world. For most items the following information is listed: brand name, manufacture, identical or similar products, chemical composition, class, appearance, properties, reaction, reference, patents, applications, and quantities used in different applications. Over 2500 commercial brands are mentioned.

Colloid Science I (Irreversible Systems). Edited by H. Kruyt, formerly of Utrecht University. Elsevier Publishing Co., New York, 1952. Cloth, 6½ × 10, 389 pages. $11.

Although the present volume is No. 1 it follows Vol. II ("Reversible Systems") which was published in 1949 and was well received by universities and industrial laboratories. The book, written in collaboration with G. H. Jonker of Phillips Research Laboratories and J. Th. G. Overbeck of Utrecht University, is meant to be a guide to the domain of colloid science for the purpose of stimulating research in the field with which it deals. Among the subjects covered are lyophobic systems, optical properties of colloid solutions, electrochemistry of the double layer, electrokinetic phenomena, kinetics of flocculation, and stability of hydrophobic colloids and emulsions.

Note: Any book reviewed in *Tappi* may be obtained through the Book Dept., Technical Association of the Pulp and Paper Industry, 122 E. 42nd St., New York 17, N. Y.

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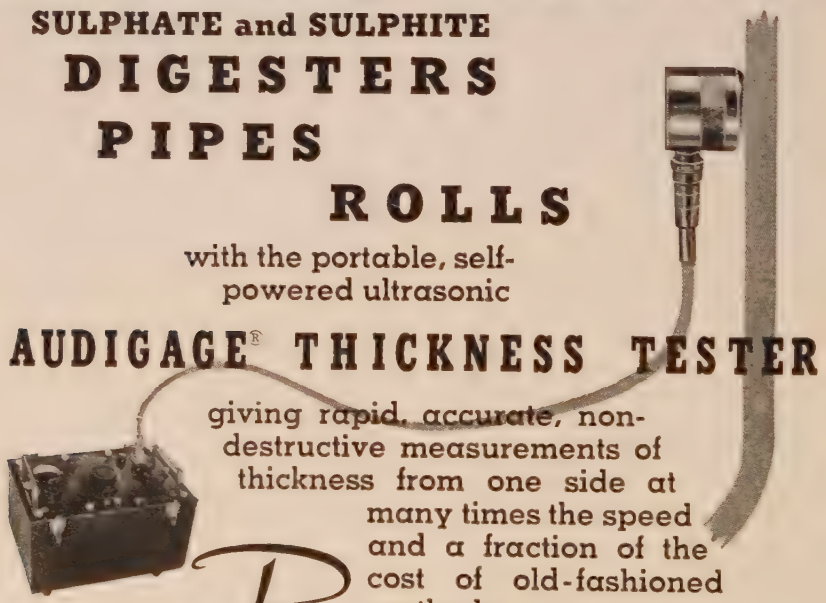

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A Chemical Study of the "Skinlike Substance" of Sulphite Pulps*

RUTH R. ROLLINSON and LOUIS E. WISE

For many years chemists have interested themselves in the relationship between the morphology of the cell wall of wood and the distribution and chemical composition of its various components. Among the investigators whose work deserves consideration are: Ritter (*1*), Lüdtke (*2*), Harlow (*3*), Trogus (*4*), Carpenter and Lewis (*5*), Lewis, Brauns, and Buchanan (*6*), Howells (*7*), and Bailey (*8*). Many of these chemists were greatly interested in components in the outer layers of wood tracheids and, following the nomenclature proposed by Lüdtke (*2*), frequently referred to such layers as the "skin system" or "skin substance." This system or "membrane" included those components of the cell wall which remained insoluble in cuprammonium hydroxide following the classical ballooning and rupture of fibrous material; or, in some instances, it included the constriction occurring when cellulosic materials were treated with quaternary bases.

Quite recently Wardrop and Dadswell (*9*) have reinvestigated the swelling phenomenon and rejected the older concept of the "skin substance." They believe that the carbohydrate fraction remaining insoluble in cuprammonium hydroxide does not represent a constant morphological or chemical fraction of the tracheids. They maintain that the composition of this fraction is probably determined by the chemical history of the pulp from which it is extracted. Furthermore, it is their belief that this so-called "skin substance" may contain portions of the primary wall, as well as some of the outer and inner layers of the secondary wall. Irrespective of whether or not the portion which forms an insoluble copper complex is resident on the surface of fibers, its presence or absence is admittedly of importance to paper pulp and dissolving pulp properties.

That the material is chemically nonhomogeneous is a reasonable working hypothesis, and our experiments give some evidence that this may be the case. An important fact that has not been stressed by other investigators is that the main portion of this insoluble material, when suitably "decopperized," is actually soluble in *cold* dilute acid and soluble in distilled water. There appears to be a component on the fiber surface (and/or possibly bordering the lumen) which forms a water-insoluble copper complex when cellulose itself passes into solution during ballooning. On the other hand, this highly insoluble material furnishes an easily soluble and readily hydrolyzable polysaccharide which is very different in its chemical composition and properties from those of the other portions of the cell wall—such as the inner portions of the secondary layer. Thus, the nonuniformity of the carbohydrates of the wall of individual cells is strongly indicated, a probability frequently stressed by Jayme (*10*). It might be well, however, to inject a conservative note at this point. The copper complex may serve to concentrate mannose units and may not be representative of the *entire* outer layer of the wood fiber.

It was the object of the present investigation to study the chemical nature of the "skin substance" from various pulps using, wherever possible, some of the recently developed hydrolytic and paper partition chromatographic techniques.

ORIENTING EXPERIMENTS

With the Lewis-Brauns-Buchanan "Skin Substance"

The copper complex was isolated by means of cuprammonium hydroxide from a coniferous sulphite pulp by Lewis, Brauns, and Buchanan (*6*). The technique of isolation (based on unpublished data of F. E. Brauns) follows. Air-dried pulp was shredded in the Abbé mill and 10 grams of this were treated with 200 ml. of a cuprammonium solution (prepared according to TAPPI Standard T 206 m-44) in a 500-ml. wide-mouth ground-glass, stoppered Pyrex bottle. The mixture was rotated overnight; the following morning the solution was diluted with an equal volume of water and rotated further for 8 hr. The mixture was then diluted with 3 parts of water and centrifuged. A pale blue precipitate was formed and washed six successive times in the centrifuge bottle with fresh cuprammonium solution, thoroughly mixing (with a mechanical stirrer) in each case for an hour and then centrifuging. In every instance, the supernatant liquor was decanted sharply before a new batch of the reagent was added. Finally, the blue precipitate was washed once with aqueous ammonium hydroxide (approximately 14%), twice with alcoholic ammonia, and finally with ether and petroleum ether. The product was filtered in every case on a tared, fritted glass crucible and dried in a desiccator containing paraffin over concentrated sulphuric acid and soda lime. The complex was decopperized by means of *N* hydrochloric acid *in the cold*. The suspension was centrifuged from a small insoluble residue (*A*) and the clear supernatant solution treated with a large excess of acetone. This treatment removed cupric chloride and caused the formation of a flocculent precipitate (*B*). The latter was washed repeatedly with acetone until free from copper salts and was then dried and hydrolyzed about 8 hr. on a steam bath with 2.5% sulphuric acid. The original floc B (prior to hydrolysis) gave a positive naphthoresorcinol test for uronic acid. The hydrolyzate of B was passed through an ion-exchange column (using Duolite A2 resin) to remove acids; this procedure partially removed uronic acids which were present among the hydrolysis products. A qualitative paper chromatogram of the hydrolyzate of B (after passing the ion exchanger) showed the presence only of mannose and glucose (with the former predominating). The presence of mannose was confirmed by isolation of mannose phenylhydrazone, m. 191–192° (Fischer m. p. block) and, in one case, by the formation of the yellow, crystalline "Blair-Wolfrom derivative," (*14*) $C_{12}H_{12}O_4N_2(OCCH_3)_4$, m. 120–121°. (An authentic sample of the latter m. 121–122° and the mixed m. p. was 120.5-122°.) The acid-insoluble residue (A) was dissolved in cold 72% sulphuric acid and the mixture diluted to 2% and hydrolyzed at about 100°. This hydrolyzate also yielded simply glucose and mannose with the former predominating.

A quantitative study was made of the hydrolyzate from B (after passing through the ion-exchange resin). The periodate oxidation micromethod of Hirst and Jones (*11*) was used. The results of triplicate determinations are given in Table I. B retained calcium salts tenaciously; these were removed only after passing an aqueous solution through the ion-exchange resin (Amberlite IR-100). This treatment reduced the ash to about 0.65%.

Table I. Analysis of Hydrolyzate of B (After Passing Through Ion Exchanger)

Sugar	*I*	*II*	*III*
Glucose, %	22.6	20.9	21.9
Mannose, %	77.4	79.1	78.1

It was found that Fehling solution precipitated an aqueous solution of the polysaccharide B in the form of a pale blue floc. This, when washed and hydrolyzed, also yielded glucose and mannose with the latter predominating. Because of this reaction, the Munson-Walker reducing value of floc B could not be determined. A preliminary hydrolysis-time study of a sample of B with 2% sulphuric acid indicated that the hydrolysis was complete within 5 hr., but that the Munson-Walker

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reducing value (in terms of cuprous oxide) did not reach the maximum required by a glucomannan (containing about 80% mannan), e.g., when 30 mg. of B was hydrolyzed 5 hr., the amount of cuprous oxide obtained was 64.3 mg. (calculated for 30 mg. of a hydrolyzed glucomannan, 75 mg. cuprous oxide) (*12*). The difference may be due to the presence of an aldobiuronic acid. However, this hypothesis is offered with great reservation. Up to the present, microanalyses for uronic acid in the unhydrolyzed B have given fluctuating results, but there is strong evidence that such an acid residue exists in the original polysaccharide. Inasmuch as the original copper complex was probably prepared from a bleached pulp, aldonic acid groupings are not excluded from B.

Summarizing, the soluble polysaccharide contains predominantly mannose units, together with subordinate amounts of glucose and what appear to be uronic acid residues.

It is of interest to note that Sherrard and Blanco (*13*) obtained a mannose-yielding polysaccharide from the cell walls of white spruce. Cross and Bevan cellulose was subjected to a protracted water extraction and this extract on evaporation gave a sirup, from which alcohol precipitated a polysaccharide (corresponding to about 12% of the cellulose). Sometimes the material was amorphous but at times microcrystals were obtained. The material was very soluble in water, insoluble in organic solvents, and had a slight reducing action on Fehling solution. Hydrolysis showed the presence of a relatively large amount of mannose (corresponding to about one third of its weight). It also gave rise to furfural (possibly because of pentose units or uronic acid groups). Further than this, the interesting material was not studied.

Studies on the Skinlike Substance from Bleached Black Spruce (Mitscherlich) Sulphite Pulp (Interlake Mill)

An analysis of the sulphite pulp for mannan showed 6.6%.

Using the methods devised by Brauns for isolation of the copper complex, the spruce pulp [20 grams (air dry)] was extracted repeatedly with cuprammonium hydroxide, using a vertically placed rotating wheel. (Brauns' second extraction of the pulp with the copper reagent was extended from 8 to 24 hr.). Dilutions were always made with water. The pale blue copper complex after drying in an ordinary desiccator weighed 0.8718 gram. This retained 6.8% moisture. Hence, the oven-dry weight was 0.8125 gram. This material yielded 30.4% ash (presumably cuprous oxide). The yield of "insoluble" polysaccharides free from copper oxide was approximately 0.568 gram (calculated). The copper complex (120 mg.) was then treated as described above, with *N* hydrochloric acid, and separated into a soluble (D) and an insoluble portion. The soluble portion was precipitated and decopperized as heretofore with acetone. The floc was then hydrolyzed with formic acid, giving a good yield of mannose phenylhydrazone. Hydrolysis of D, followed by paper partition chromatography, showed glucose and mannose, the latter predominating. The soluble portion D (of a similarly prepared decopperized polysaccharide) was precipitated with acetone and washed successively with acetone, alcohol, and ether. The carbohydrate was dissolved in water and sufficient 10% sulphuric acid was added to bring the acid concentration to 5%. Hydrolysis of the material was effected by heating. The mixture was then passed through a Duolite A2 column to remove acids, the solution was concentrated to a sirup, and chromatographed, using a mixture of pyridine-butanol-water. Results are shown in Table II.

Table II. Analysis of Hydrolyzate D from Black Spruce Mitscherlich Pulp (After Passing Through Ion Exchanger)

Sugar	*I*	*II*	*III*	*IV*
Glucose, %	22.1	22.1	21.6	14.6
Mannose, %	77.9	77.9	78.4	85.4

Sample IV has been included, although the results are in some doubt. It is evident that the ratio of the two sugars in D is substantially the same as that shown in Table I.

When the isolation of the insoluble copper complex was repeated, using the same sulphite pulp and what appeared to be conditions similar to those given above, varying yields were obtained. This lack of reproducibility has not been fully explained; it may be due to differences in the rate and length of stirring the pulp with the reagent, in the age of the cuprammonium solution, in the temperature, *et. al.* These fluctuations are being studied further. It was shown that the (Lewis-Brauns-Buchanan) copper complex which is quite insoluble in water, is partially soluble in the cuprammonium reagent.

Studies on Aspen Sulphite Pulp

This sulphite pulp was prepared from trembling aspen. When treated repeatedly with cuprammonium hydroxide, as above, only a minute amount of residue was obtained, which appeared to be completely insoluble in cold *N* hydrochloric acid. No floc was obtained from the supernatant aqueous acid and, on attempted hydrolysis overnight with 3.5% sulphuric acid, the insoluble residue apparently failed to dissolve or to hydrolyze.

Studies on Cuprammonium-Insoluble Residues from Slash Pine Alpha-cellulose

This sample of alpha-cellulose was prepared from a slash pine holocellulose by treating the latter with potassium hydroxide under nitrogen. Two samples of this cellulose were treated with cuprammonium hydroxide but somewhat differently in either case (E and F).

E. 10 grams (air dry) were treated by the Lewis-Brauns-Buchanan procedure.

F. 10 grams (air dry) were similarly treated, except that cuprammonium solution, not water, was used in the dilutions.

The copper complex from E (about 150 mg.) was treated with *N* hydrochloric acid, giving a soluble and an insoluble fraction. The soluble portion was flocked out by the usual acetone procedure and, on hydrolysis, showed the presence of galactose, glucose, and mannose (the last predominating). A small amount of disaccharide may also have been present. The insoluble fraction on hydrolysis and chromatographing indicated the probable presence of galactose, mannose, and glucose.

The copper complex from F (total weight from 10 grams alpha-cellulose about 0.0986 gram) with hydrochloric acid gave 71.1 mg. insoluble material (F_1) and 7.8 mg. (F_2) floc from the soluble material. F_1 on hydrolysis gave galactose (strong test), glucose, mannose, arabinose, and rhamnose. F_2 gave a *strong test for mannose*, a definite test for galactose, and possibly glucose and xylose. (All these results are based simply on paper chromatograms and would require further chemical confirmation.)

The work is being continued using other pulps.

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Instrumentation Studies. LXXVI. A Study of Photoelectric Instruments for the Measurement of Color: Reflectance and Transmittance

X. *The "Color-Eye" (P.P.G.-I.D.L.)*

J. A. VAN DEN AKKER, LEONARD R. DEARTH, O. HARRY OLSON, and WAYNE M. SHILLCOX

This report presents the results of a thorough study of the Color-Eye (Instrument Development Laboratories, Inc.); the study was conducted with the aim of ascertaining the potential suitability of this three-filter colorimeter for color matching and color control in the paper industry. The introduction describes the uses of colorimeters and discusses the requirements to be met by such instruments employed in the paper industry. The discussion includes the fact that all matches in the paper industry are "metameric" in varying degrees; it is also pointed out that, to be reliable, a filter colorimeter should not indicate a match when a color difference may be seen, that it should not indicate an appreciable mismatch when a color difference cannot be seen, and that it should indicate the proper direction and magnitude of a color difference. Following a brief description of the Color-Eye, observations on various instrumental characteristics are given. The instrument possesses excellent operational characteristics (ease and speed of use, stability, sensitivity over a large range of light intensity, ruggedness, compactness, etc.); the instrumental results are somewhat frequency-dependent, and special arrangements are made where a constant-frequency line is not available; line-voltage dependence is nil. A significant weakness of the instrument is the fact that the interior of the integrating sphere, to which the specimen and standard are exposed, is quite hot; in consequence, a reference paper (used as a standard of color similar to that under control) may not be used, because the fading is rapid. The elevated temperature caused an appreciable change in reflectance of the "Vitrolite" standards over an extended period of time. (New models may be furnished with means for external illumination of specimen and standard, with geometries different from that of the integrating sphere and, of course, it is possible that the heating effects may thus be minimized.) The photometric accuracy was tested and found to be poor. Possible reasons for the photometric errors are given in the hope that the discussion might aid the manufacturers in improving the design of the instrument. The performance of the Color-Eye in color matching was studied through spectral transmittance determinations, using alternately the Color-Eye and the G.E. recording spectrophotometer (GERS), in which several families of dye solutions and several glass color filters were employed. Spectral reflectance determinations were made on a collection of colored papers and for nine series of papers involving color differences typical of those encountered in the industry. ICI tristimulus values and trichromatic coefficients were determined from both sets of instrumental data and compared. Absolute errors are given for those persons interested in the possibility that the Color-Eye might serve as a rapid analog computer, to replace the somewhat arduous work of obtaining a spectrophotometric curve and integrating to determine the ICI tristimulus values. The absolute errors are generally quite large. Those observed in the tristimulus values for the solutions and glass color filters are discussed in some detail, and appear to be clearly explicable on the basis of certain instrumental imperfections. The manufacturers of the Color-Eye emphasize the use of their instrument in the measurement of color differences (a matter of primary importance in the production control of color). For the purpose of the present report, differences in luminous reflectance are ignored, and the comparisons are based on intervals or segments on the ICI chromaticity diagram. Techniques for comparison are discussed and used; it is shown that the Color-Eye indications of the nature of color differences (in terms of direction and magnitude of segments on the chromaticity diagram) are sometimes nearly right but that, in a fair proportion of the cases, the errors vary from small but significant to quite large. As might be expected, the errors are largest where the degree of metamerism is greatest, but there are cases where the errors are large in spite of the use of one-component colorants. The report terminates with a reiteration of the needs in the paper industry for rapid colorimetry, of the fact that color matches in the paper industry are metameric (the color man is not in a position to know how metameric), and with a plea that manufacturers of filter colorimeters analyze the spectral requirements of filter colorimeters more thoroughly, with the objective of producing improved instruments.

Color-measuring instruments employed in the pulp and paper industry are of two types: these are spectrophotometers (either monochromatic or abridged) and colorimeters. Instruments of the first type yield spectral reflectance or transmittance curves and are indispensable in research and in certain kinds of industrial control. Of all the instrumental methods known, the spectrophotometer, when used in conjunction with suitable integrating equipment, is the most accurate and reliable for the evaluation of color and color difference. Furthermore, the spectral reflectance and transmittance data yielded by a spectrophotometer enable one to obtain some understanding of the physical factors underlying a color or a color difference. However, for the purposes of color control on the paper machine, a spectrophotometer is believed by many people to be too slow in the rate at which it can yield information and unduly elaborate for the function of process control. Hence, over the years, three-filter photoelectric colorimeters have been de-

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veloped for color matching. The undisputed advantage of these instruments of the second type is that they yield three numbers supposedly corresponding to the ICI tristimulus values X, Y, and Z, in a very short period of time. The claim is usually made that, where colorimeters are used to measure small color differences (of the kind normally encountered in manufacturing processes), the color differences will be measured reliably and, because such instruments are very sensitive, the reader of technical literature published by the instrument manufacturers is often left with the impression that colorimeters "see" color differences that are smaller than can be detected by the human eye. There is little question concerning the sensitivity of colorimeters; it is simple to demonstrate that a modern colorimeter is capable of measuring incredibly small differences in variables which are functions of reflectance or transmittance. *The important question is whether those functions are the proper functions for the reliable evaluation of color differences, for it may be shown that, if the functions are not correct, a colorimeter may indicate an appreciable color difference when the normal eye sees a match, and a match arrived at through the dictates of the colorimeter may appear to be a serious mismatch to the normal eye.*

The manufacturers of three-filter colorimeters hasten to say that industry should avoid metameric color matches (i.e., those which match under one illuminant, such as daylight, but fail to match under a quite different illuminant, such as tungsten-filament light). This is a good admonition but papermakers are often forced to produce color matches which are rather strongly metameric. Actually, except by rare chance, *all matches in the production of paper—even where the furnish is controlled with regard to the kinds of pulp and dyestuffs—are metameric in various degrees.* (Two colors form a nonmetameric near-match only in the highly accidental case in which they differ only in the luminous reflectance Y for all illuminants.) A potential user of a colorimeter should, then, be very concerned with the question as to whether the instrument will properly indicate the nature of the typically metameric mismatches encountered daily in the production of paper. Numerous persons have said, or implied, that a colorimeter will measure small color differences reliably if (1) the difference is small, and (2) the reference standard has color like that under control. This seems, *on an intuitive basis*, to be correct. Analyses of real situations with past three-filter colorimeters have failed to justify these claims, and past instrumentation studies have failed to support them. Nevertheless, it is theoretically possible for photoelectric colorimetry to be developed to the accuracy at which it will simulate reliably, in many situations, the average normal human eye, and be more sensitive than any existing eye. Hence, research and development leading to suitable multiple-filter colorimeters should be encouraged.

Part I of this series of reports (*1*) presents a detailed discussion of the principles of photoelectric color instruments and also a condensed presentation of the ICI system of color specification. Parts IV and VIII (*2*, *3*) will probably be of interest to the reader because of their intimate connection (through fundamentals) with the present work. The reader interested in the theory of three-filter photoelectric colorimeters may wish to consult a discussion of the limitations of the best physically realizable three-filter colorimeter (*4*).

The Color-Eye, developed by the engineers of the Glass Division Research Laboratories of Pittsburgh Plate Glass Co. and the Instrument Development Laboratories, Inc., is the subject of the present study. Although numerous color instruments have been studied in past instrumentation projects, it was deemed to be of interest to add Color-Eye to the program, because this instrument possesses certain definite merits such as ruggedness, simplicity in operation, stability, adaptability to light level, and sensitivity. An instrument was loaned for the purposes of the study through the kind cooperation of the manufacturers, the Instrument Development Laboratories, Inc., Needham Heights, Mass.

DESCRIPTION OF THE COLOR-EYE

In view of the fact that the Color-Eye has been described by the manufacturers (Instruction Manual I.M. 5002), only a brief description of the instrument and its functions will be given here. A photograph of the instrument studied in this investigation is shown in Fig. 1. The geometry of illuminating and viewing

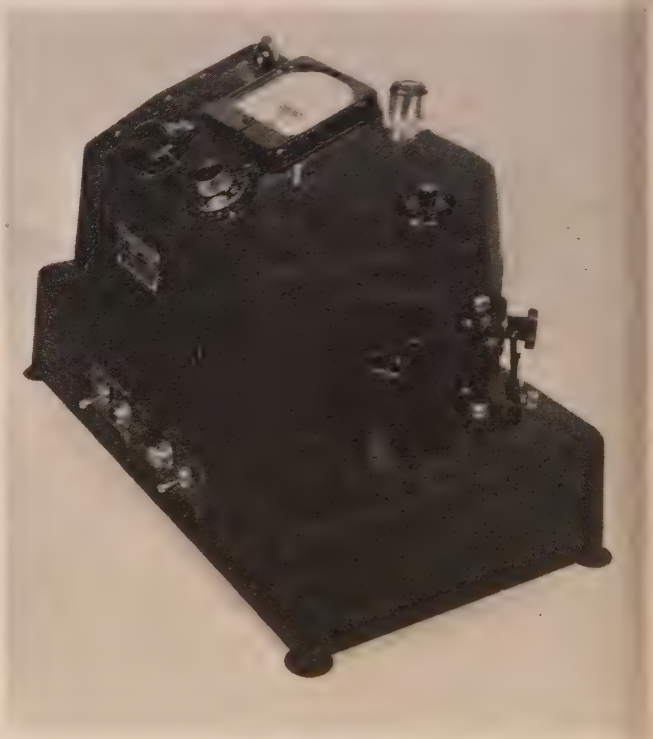


Fig. 1. Photograph of the Color-Eye

is essentially diffuse illumination, with viewing over a small range of angles at 40°. The specimen and standard are illuminated and viewed simultaneously by provision in the integrating sphere wall of specimen and standard ports, over which the specimen and standard are held by conveniently manipulated holders. Illumination is provided by a tungsten-filament lamp mounted in the center of the sphere; stops prevent direct illumination of the specimen and standard. The lamp is operated at a color temperature of 2848°K., and provision is made for measuring the lamp voltage.

That portion of the radiation accepted by the viewing system which is specular (mirror-like) originates in a small portion of a "specular wheel," which may be so adjusted as to emit (1) no light ("nonspecular" position), or (2) light from a reflecting portion. In most work the specular wheel is set in the nonspecular position. This wheel is employed, e.g., in studies involving the effect of gloss on the color.

The photoreceptor of the light-measuring system is a photomultiplier tube (1P22) which, because of its extraordinarily high sensitivity, permits the use of an ingenious electronic computing system which automatically adjusts the sensitivity of the over-all photoelectric-electronic system in such manner that the meter deflection is proportional to the percentage difference between the specimen and the standard (with regard to weighted average reflectance); the meter reading is independent of the light level over a range which is said by the manufacturer to be 5000 to 1. This and the stability of the device are perhaps the most distinguishing features of the instrument. According to the manufacturers' suggested method for the routine production control of color, the operator deals with the deflections of the meter; these can be read from a high-sensitivity range (91 to 110%) or a low-sensitivity range (70 to 140%), with 100% (according to the manufacturers' claims) corresponding to exact match between the specimen and standard, but actually corresponding to equality in the instrumentally weighted reflectances of the specimen and the standard. A photometer is built into the instrument so that one may obtain reflectance or transmittance ratios which are substantially less than 100%. This is a "micrometric slit" located in the standard beam; it is opened and closed by means of a "Duodial" linked to a Brown and Sharpe micrometer. When this photometer is employed, the Duodial is adjusted to obtain zero deflection (reading of 100%) of the meter. At this point the flicker arrangement causes the photomultiplier to receive fluctuating light beams from the specimen and from the combination of standard and photometer slit which are of equal intensity. The reading on the Duodial gives the reflectance or transmittance. A slide for liquid cells or optical filters is provided at a point between the integrating sphere and the light-measuring system.

In the Model B Color-Eye now in production, the integrating sphere may be readily removed for any of several purposes, one of which is to facilitate the use of special external illuminants; another is comparison of light sources, etc.

The spectral characteristics of the Color-Eye may be readily modified through the use of two filter wheels, mounted in series at a point just ahead of the condensing lens for the photomultiplier tube. One of these wheels enables the operator to change the source, in effect, from ICI Illuminant A to an approximation of ICI Illuminant C. The other contains the three "tristimulus" filters, each of which has been designed to result in a spectral product of phototube sensitivity, filter and glass optics transmission, and integrating sphere constant approximating the corresponding tristimulus function for the ICI Standard Observer. This is done in a manner similar to that employed in other and earlier three-filter colorimeters. As has been done in the past, the blue filter used to obtain Z (the "blue" tristimulus value) is made to serve double duty and is employed to obtain that portion of X arising in the $\bar{x}$ tristimulus function between 400 and 500 mmu.

Claims Made or Implied for the Color-Eye

The instruction manual for the Color-Eye is detailed and is enthusiastically written. Some of the claims made for the instrument are important and are undoubtedly well founded; they relate to rudimentary instrumental characteristics, such as ease of use, speed of operation, stability, and sensitivity. As indicated earlier in this section, the ability of the instrument to accommodate itself to an extremely great range of light level and yet indicate the percentage difference between the specimen and standard (with regard to reflectance) is a distinguishing feature.

There are, however, few conservative remarks to be found in the instruction manual. Undue emphasis seems to have been placed on the inability of the human eye to detect changes in luminous reflectance or transmittance, as against the high sensitivity of the Color-Eye. The remarkable sensitivity of the normal eye to very small changes in chromaticity (i.e., in hue and saturation) does not receive adequate attention. The matter of how to estimate color differences from instrument readings is presented in Section IV of the instruction manual, but the implications of the methods presented there (including the Nickerson method) with regard to the requirements to be met by a filter colorimeter receive practically no attention. A reader of the instruction manual receives the distinct impression, at several points, that, with proper operation, the Color-Eye will measure color difference with exactness. Some of these impressions are created by statements like the following: On page 4, with regard to a test for an instrumental adjustment, it is stated: "This test must be made with extreme care, as the zero shift error you are checking is one tenth the amount of difference you can see visually!!" On page 5, with regard to the point that the operator need not standardize exactly when employing the deflection method: "After all, an error of 0.5% in 8.6%, is about 0.05% or $^1/_{20}$% in 1%!! This is about $^1/_{30}$ of the difference you can see." This last remark, although the intention was to warn against "fussiness" in calibration, would lead the reader to believe that, even when used without perfect adjustment in the calibration procedure, the instrument can see color differences about thirty times smaller than those humanly detectable. One of many questions that might well be raised at this point is this: "If the Color-Eye indicates that a match has been achieved between specimen and standard—to within the small limits of instrumental detectability—may there not be an easily perceptible color difference to *all or nearly all* observers having normal color vision. for the same source of illumination, and for similar geometries of illuminating and viewing?" On page 5, the last statement implies that the Color-Eye is capable of pronouncing perfect matches: "Last But Not Least, return the duodial to 100.0%. If you forget to do this, perfect matches will be at 108.6% instead of 100%!!" The point in question here is not that there might be difficulty in arriving at a "perfect match," but that if the instrument sees a match as perfect, human observers

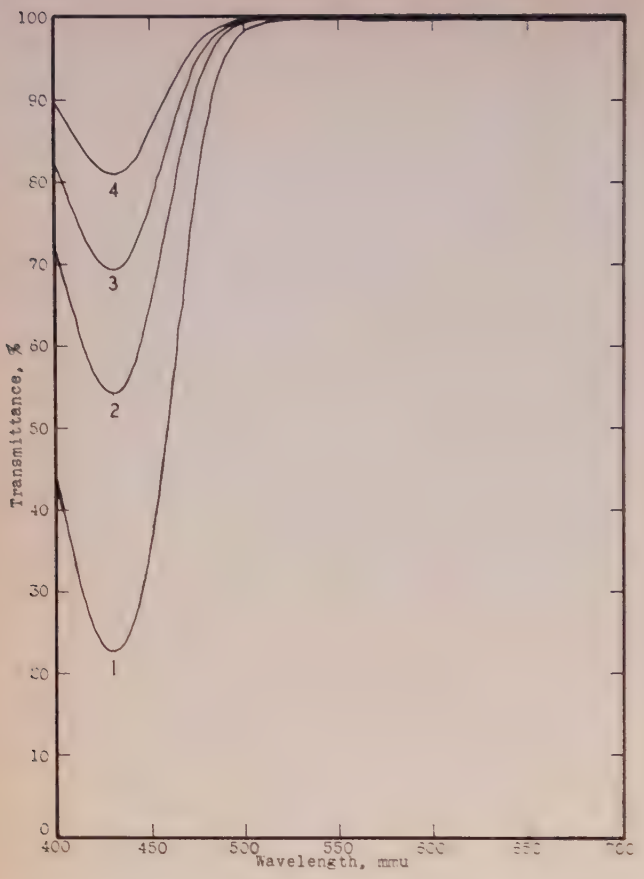


Fig. 2. Spectral transmittance of aqueous solutions of Auramine dye (GERS)

would also see the match as perfect. As discussed in Section II of Part VIII of this report (*3*) (to which the reader should make reference for clarification), a three-filter colorimeter is "color blind," in the sense that, like an anomalous trichromat, the instrument will sometimes pronounce a match as perfect when a color difference may be clearly seen by an observer with average normal vision and, vice versa, the opposite situation will sometimes be encountered.

These considerations are obviously of the utmost importance to anyone in the paper industry who is contemplating the use of any three-filter colorimeter for careful color control, with regard to control within a run and to long-range control to meet established mill standards, and also as a general aid in meeting new specifications of color, conducting special studies such as fading, two-sidedness of color of paper, etc. Paper sheets usually present a field of colorimetric comparison favoring very good discrimination of color differences: (1) large field of illumination, (2) good level of illumination, (3) binocular vision, (4) an exceedingly fine line of division between the areas to be compared and (5), usually, smooth surfaces. Accordingly, experienced color men in the industry can discern very small color differences and are quite critical.

GENERAL OBSERVATIONS

In all experimental observations, the instrument was employed only after the recommended warm-up time, and all observations were made in accordance with recommended procedures. The material presented in this section relates primarily to rudimentary instrumental variables. Observations relative to the performance of the Color-Eye as a color difference indicator are presented in the next section.

Operational Aspects

When used (as recommended for ordinary color control work) as a deflection instrument, the Color-Eye is, in our experience, unexcelled in speed of measurement, ease of manipulation, ease of reading, stability (on a well-regulated power line), and sensitivity. The instrument is small, rugged, easy to adjust, and relatively foolproof.

Dependence on Line Frequency

The Color-Eye is connected to a power line through a constant-voltage regulator. Such devices are very effective when the line frequency is constant, but disturbing fluctuations may be observed where (as in many mills) the power lines are not well controlled with regard to short-time frequency variations; this remark holds for electrical and electronic instruments generally. The frequency-dependence of the Color-Eye was observed to check the statement by the manufacturers that the control voltage will change 2.5% for a 1% frequency change and also to explore the influence of line frequency changes on instrumental readings. The manufacturers request users of the Color-Eye to give them details concerning the available power lines, in the event that a frequency-stable supply is not available, so that recommendations may be made.

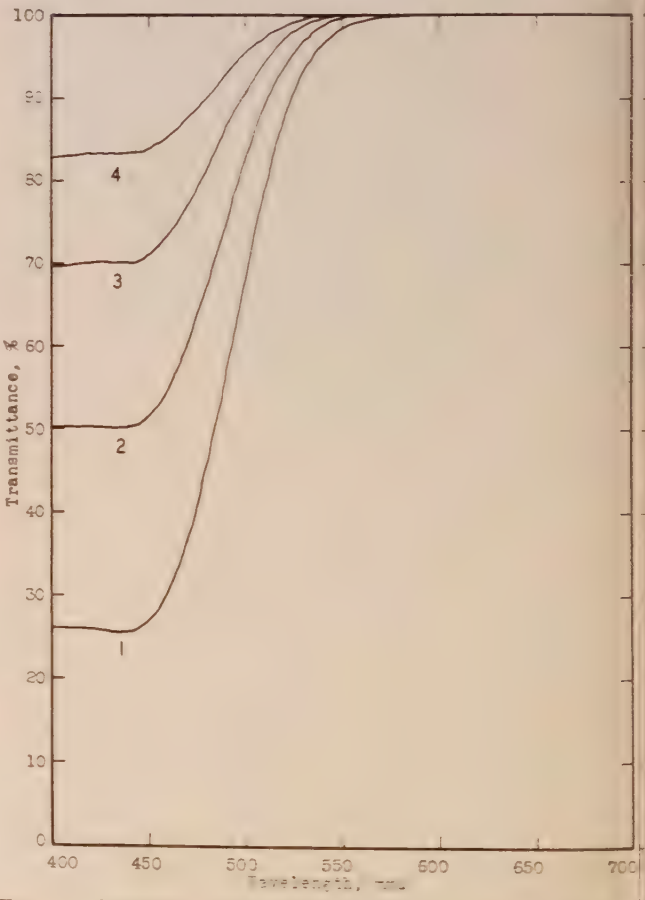


Fig. 3. Spectral transmittance of aqueous solutions of Chrysoidine dye (GERS)

The experimental, variable-frequency power line at the Institute was employed at constant voltage to obtain the results given Table I.

Table I. Frequency-Dependence of the Color-Eye

Frequency, cycles/sec.	*Lamp voltage*	*Null point, %*	*Deflection, "Hi" scale, %*	*Deflection, "Lo" scale, %*
58	8.2	99.7	107.0	141.0
59	8.6	99.7	107.8	142.5
60	8.85	99.7	108.5	143.1
61	9.2	99.6	109.7	144.3
62	9.6	99.6	Off Scale	145.2

The data given in the last two columns of Table I are suggestive of the maximum errors that might be observed through the use of the deflection method (at any given frequency). They were obtained by placing the two "Vitrolite" standards with which the instrument was equipped over the standard and specimen ports and setting the Duodial at 92.0% (resulting in data in the forth column), and at 70.0% (resulting in the data in the last column). The dependence of the lamp voltage on the frequency is essentially that given by the manufacturers (2.5% per 1% frequency change), whereas the error in the deflection is about 1% reflectance for one cycle per sec. The null-point was very stable, the change being about 0.1 for 4 cycles per sec.

A limited number of readings of X_{CE}, Y_{CE}, and Z_{CE} for colored papers showed that the results were nearly independent of frequency over the range of 58 to 61 cycle per sec. There were occasional changes of the order of 0.5%; no doubt colored materials could be found for which the frequency error would have been larger. The data presented in this section illustrate the need for the warning by the manufacturers to obtain recommendations from them where a frequency-stable line is not available.

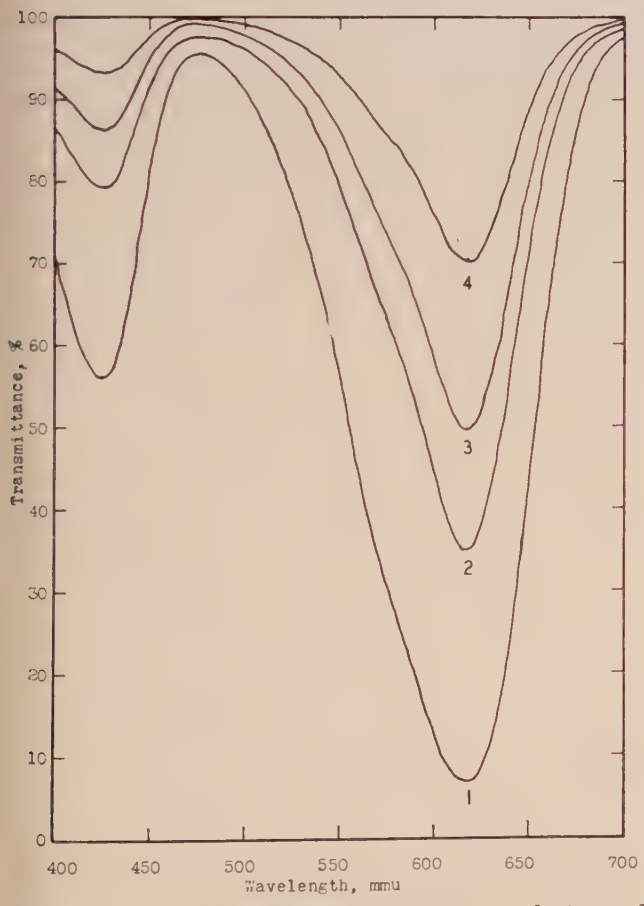


Fig. 4. Spectral transmittance of aqueous solutions of Victoria green dye (GERS)

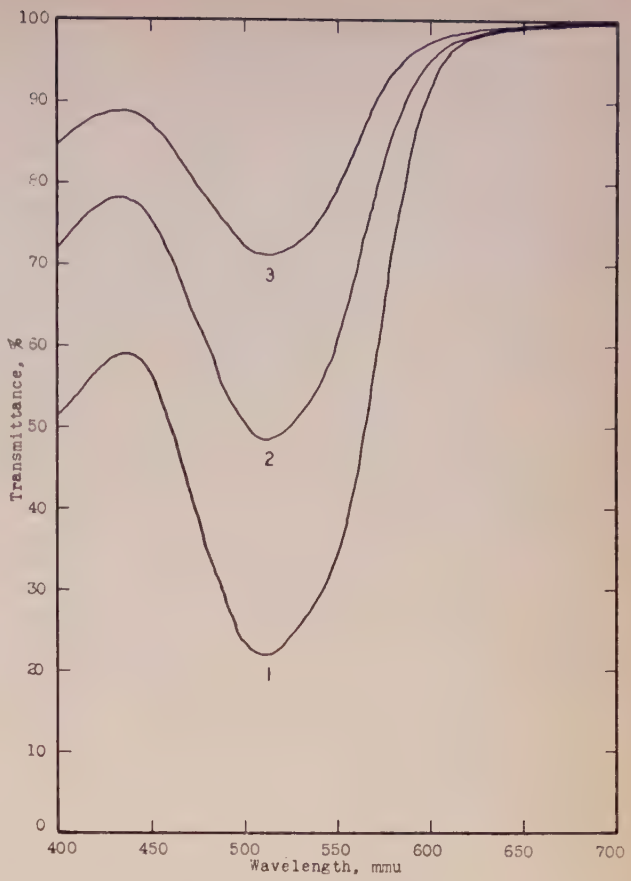


Fig. 5. Spectral transmittance of aqueous solutions of Fast red 8BL dye (GERS)

Dependence on Line Voltage

With the frequency constant at 60 cycles per sec., the line voltage was varied from 100 to 120 volts, with the result that there was no appreciable change in lamp voltage or instrumental sensitivity.

Linearity

The photometric systems of most color-measuring instruments exhibit the largest errors near midscale because procedures ordinarily assure that the scale is correct at 0 and 100%. The linearity error in an instrument designed for measuring color difference is unimportant if the reflectance (or transmittance) of the reference standard is closely similar to that of the specimen. However it often happens in a paper mill that the reflectance of the paper in production has a reflectance of the order of one half that of "working standards." Hence, it was felt desirable to check the linearity of the Color-Eye at a few points not far from midscale.

In a first attempt, four sooted wire cloth screens were employed. With the Color-Eye set for measurements of Y_{CE} and the General Electric (Hardy) recording spectrophotometer (GERS) set at a wavelength near

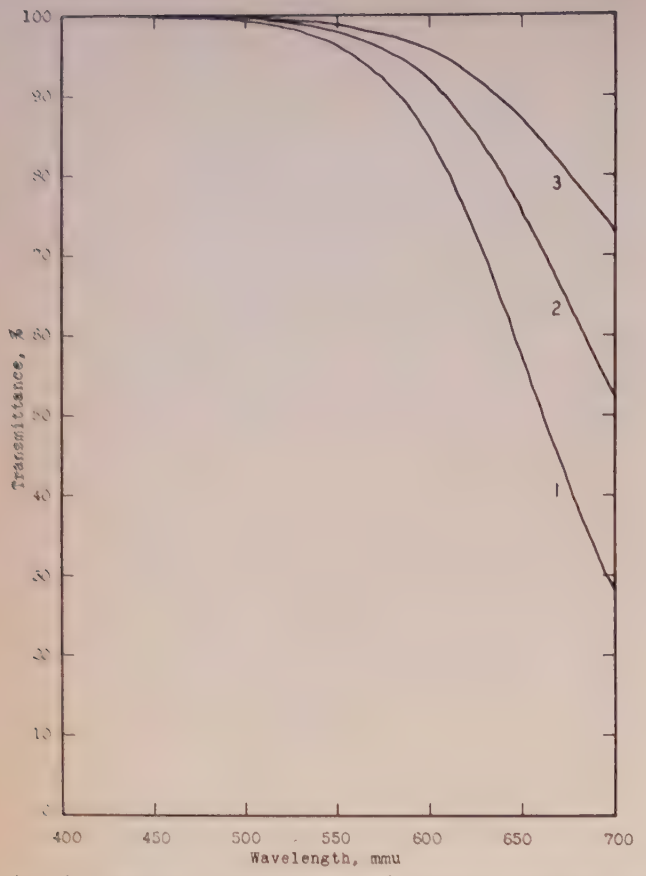


Fig. 6. Spectral transmittance of aqueous solutions of copper sulphate (GERS)

555 mmu, the transmission of a screen was measured alternatively with the GERS and the Color-Eye. Six measurements were made with each screen. The screen was handled in such manner that almost the same zone was employed in the two instruments. In each case, transmission was measured with the Color-Eye using the "micrometer slit" (controlled through the Duodial). The photometer of the GERS is routinely set with matched standards, and its linearity was in this case checked by special means employing an auxiliary zero-resistance blocking-layer photocell arrangement with a galvanometer that is known to be accurately linear, through the medium of sooted screens. After placing a screen in the GERS and making a determination of transmission, the flicker motor was stopped and set in a position to yield maximum light flux through the screen and the specimen port of the sphere. The GERS lamp was operated at constant voltage by means of a Model 150 Sorensen regulator and a step-down transformer. A selected blocking-layer photocell, used in a zero-resistance circuit with a galvanometer of tested linearity, was placed over the specimen port of the GERS, and a reading was taken without disturbing the screen. The screen was then removed, and the reading (nearly full-scale by prior adjustment) was obtained to calculate the transmission. Transmission values at 38.0, 40.6 and 60.0% on the GERS scale differed from those obtained with the special arrangement by −0.05, +0.05, and +0.15%, respectively. Earlier measurements on the accuracy of cutting of the "tangent-squared" cam of the GERS had shown that the photometric system of the GERS should be accurate to within 0.2%. The results of the comparison between the GERS and Color-Eye transmissions were (Color-Eye transmissions given first): (20.6 − 19.9 = 0.7); (39.1 − 38.1 = 1.0); (42.4 − 41.1 = 1.3); (62.4 − 61.2 = 1.2).

Errors of the magnitude indicated in the foregoing would be regarded as serious in certain circumstances, as in an organization where two or more instruments are used, and where the diversification of products is such that the micrometric slit would be employed. The screens were not highly uniform and, although an effort was made to use the same area in each instrument, note was made of the fact that the beam size in the Color-Eye at the point where transmission measurements are made is small. Also, scattering of light by the screens, although small, would have different relative effects in the two instruments. In consequence, the Color-Eye readings were more variable than is considered desirable in this type of work. The maximum, average (in italics), and minimum readings for the Color-Eye data presented above were (21.7, *20.6*, 20.0); (40.1, *39.1*, 38.0); (43.6, *42.4*, 41.5); and (62.8, *62.4*, 62.2). A more accurate comparison was sought through the use of a special "standard screen" prepared in our laboratories a number of years ago. This screen is comprised of a regular array of holes, each with a diameter of 0.1090 in. The edge of each hole had been beveled during the preparation of the screen, so that the correction for the finite thickness of the edge of each hole would be small. By calculation and measurement, the transmission of this screen for nearly parallel light is, very nearly, 42.4%.

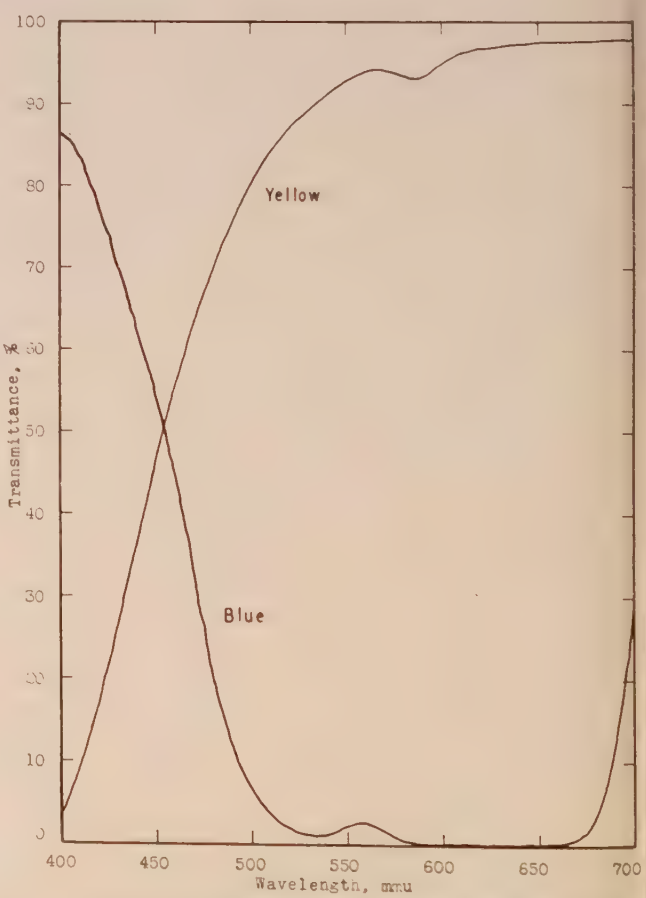


Fig. 7. Spectral transmittance of blue and yellow glass color filters (GERS)

The standard screen was too large and too coarse to place in the transmission compartment of the Color-Eye and, hence, the Color-Eye was arranged for operation with a pair of external light sources. Two 75-watt, 120-volt Mazda lamps having diffusing bulbs were connected in parallel to the same power line, and arranged so that one centered on the optical axis passing through the standard port and the other centered on the specimen axis: each lamp was about 2 ft. from the corresponding sphere port. All light from these lamps was screened excepting that issuing from holes in black paper sheets placed in front of the lamps; these holes were a little larger than the zones from which light was accepted by the optical system of the Color-Eye. The room was dark, and stray-light effects were negligible. With the Color-Eye in operation and the meter at its null deflection (100.0% on the scales) with the micrometric slit set at 100.0, the standard screen was held in the specimen beam and the Duodial was turned to obtain the null deflection. The standard screen was held at a point about 1 in. in front of the lamp on the specimen side and, for each reading, the screen was held in a slightly different position. All readings were nearly the same, at an average value of 44.6%. This determination indicates an error of +2.2% at 42.4%, as compared with the apparent error of +1.3% at 41.1% determined earlier with the sooted wire screens. There is a substantial discrepancy between the results of these two determinations, which may be partially the result of the uncertainty in the results obtained with the wire screens.

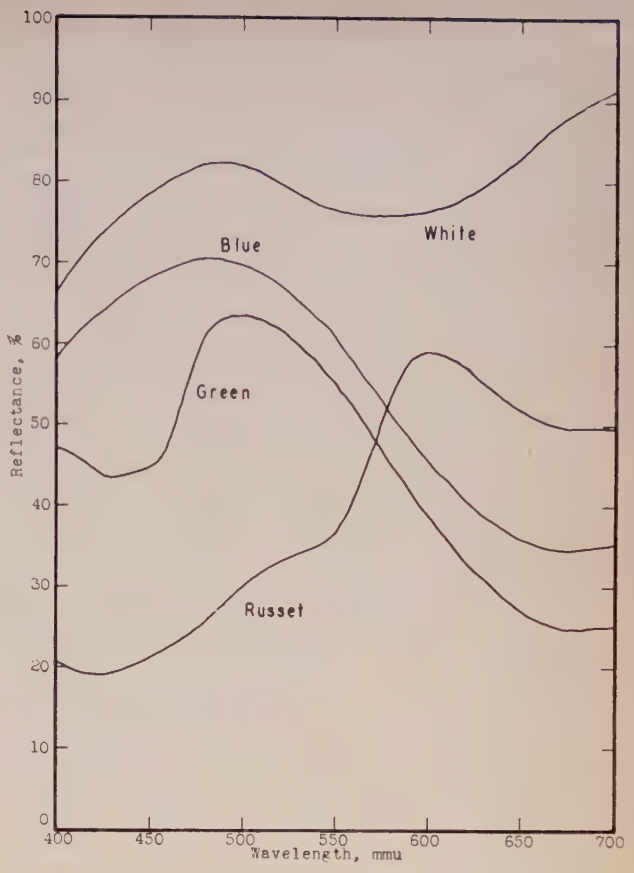


Fig. 9. Spectral reflectance of white and other bond papers (GERS)

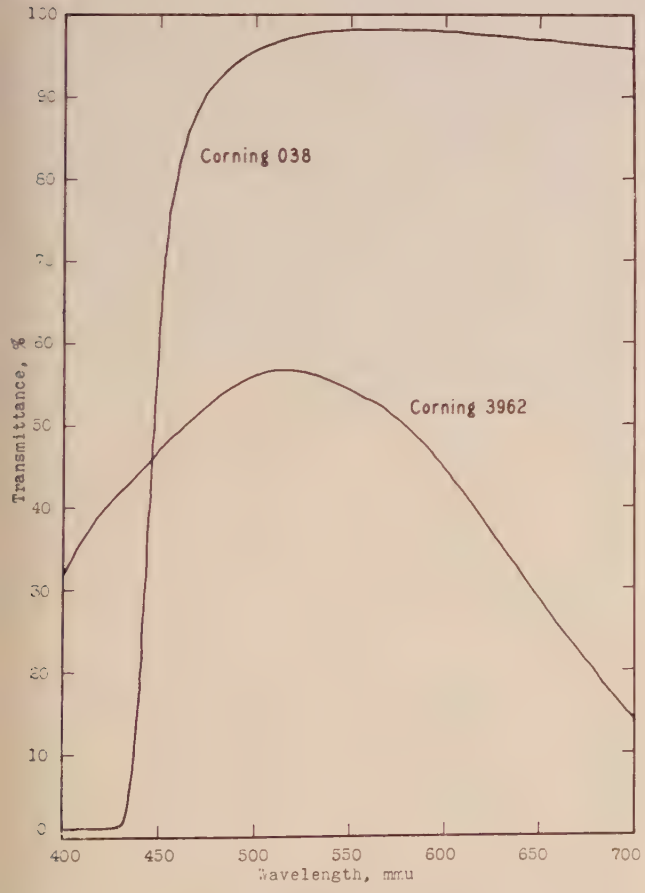


Fig. 8. Spectral transmittance of two Corning glass filters (GERS)

The uniform illumination of the micrometric slit would seem, superficially, to ensure good photometric accuracy. However, imperfections of the optical system can result in error as readily as though they existed *between* the light source and the slit, instead of beyond the slit. The system could not be examined directly, but inspection along the standard axis with the lamp of the Color-Eye turned off and a flashlight directed into the system revealed contamination of the optics. It seems likely that an uneven distribution of particles of dust and other materials on the optical components would result in error. If this is actually a significant source of error, one would expect the magnitude of the error to change with time, as dust and debris collect on the surfaces of the optics. It is commonly observed that lenses, prisms, and other optical components in instruments that are enclosed as effectively as are the parts of the Color-Eye gradually accumulate surface films and fine dust. It is possible, then, that the photometric error is time-dependent.

A possible source of error lies in the consideration that the point-to-point sensitivity of the surfaces of phototube cathodes is not uniform. If the optical system of the Color-Eye is not such that the actual distribution over the cathode of light after passage through the *partially opened* micrometric slit is identical with that of light after passage through the zeroing slit, there will be a photometric error. The magnitude of the error resulting from inequality of the distributions may be determined only through a careful study of the optical system of the Color-Eye.

Temperature of the Sphere and Fading of Standards

The warmth of the integrating sphere of the Color-Eye prompted a query relative to the possible effects on materials, especially papers, of elevated temperature and light. Past studies have shown that the fading of paper by light may be accelerated by heat and, of course, heat has a well-known "aging" effect on paper. The effect on paper specimens under momentary test is clearly negligible, as each test specimen is in contact with the sphere for only a fraction of a minute. However, it is natural to expect that users of the Color-Eye in paper mills would wish very much to compare the color of paper in production with that of "standard papers." The latter would be used in the standard position. Any one standard paper might well be left over the standard port for a matter of hours or, alternatively, any given reference paper might be used briefly at times but be exposed for a substantial integrated time.

Using a thermoelectric surface pyrometer, it was found that the outside surface of the integrating sphere, near either port, was at a temperature of about 130°F. In order that at least a crude measure might be made of the inner temperature (to which the surface of the standard and specimen are exposed), the pyrometer was placed in the hole of a one-hole rubber stopper, and the latter was carefully introduced into a port, so that the thermojunction was just inside the sphere. In this position, the pyrometer read 150°F.

In an attempt to evaluate the influence of the heat and light on the color of paper, 2-hr. exposures were made of a cherry, a gray, and a blue sulphite bond, and a 2.75-hr. exposure was made of a white bond. Spectrophotometric (GERS) curves were obtained before and after the exposures; of course, the determinations were for the same specimens, the same backing, and the same area of test. The curves are given in Figs. 17, 18, 19, and 20, and the Color-Eye determinations are included in Table VI.

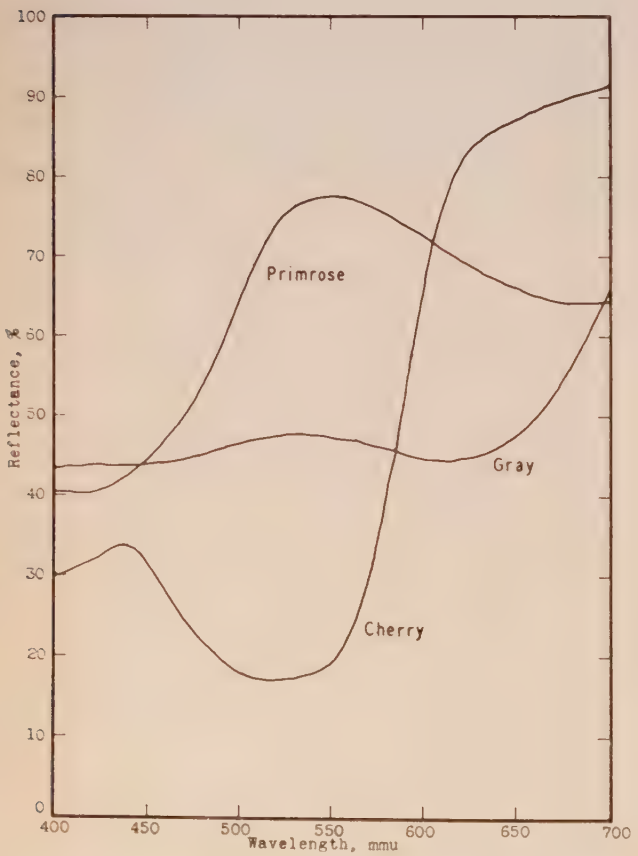


Fig. 10. Spectral reflectance of colored bond papers (GERS)

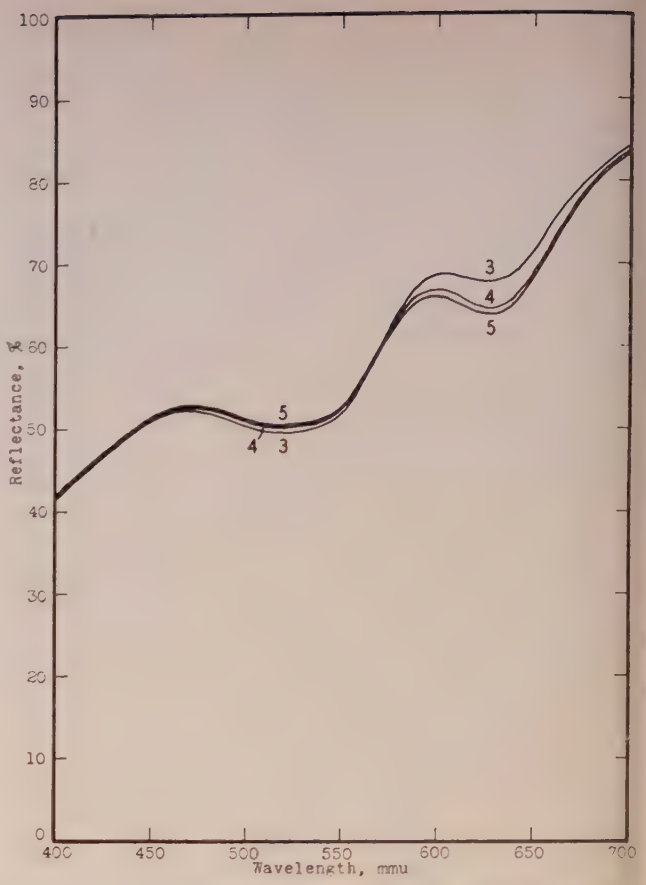
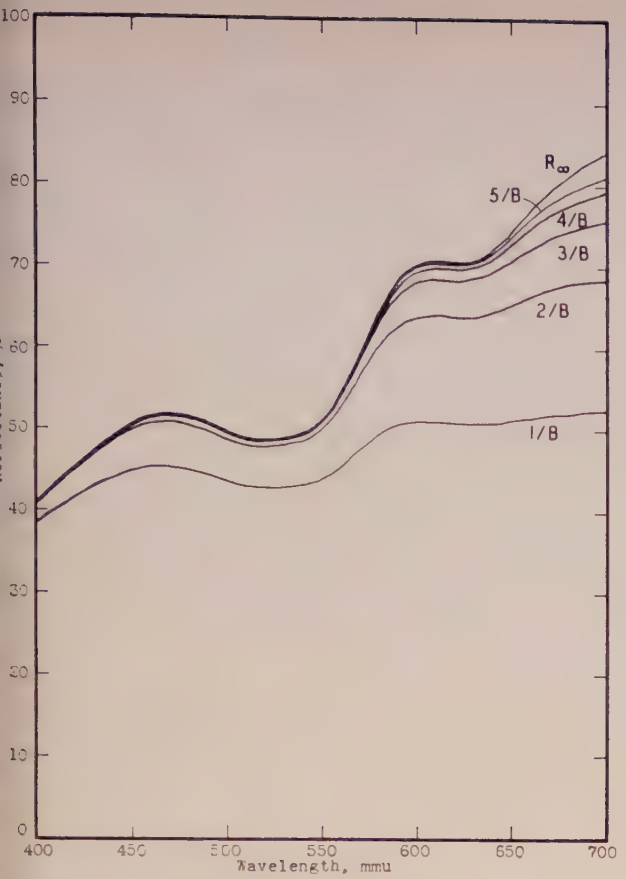


Fig. 11. Spectral reflectance of a series of pink handsheets (GERS)

To those persons who are unfamiliar with the relationships between differences in spectrophotometric data and color differences, it should be pointed out that the differences displayed in Figs. 17 to 20 are not necessarily small. A distinct difference in color can be seen on comparing the faded and unfaded cherry bond; in the case of the white and gray bonds, a central circular portion of diameter the same as that of the sphere ports can just be discerned in the exposed sheets; the change in the blue paper is just noticeable. However, the duration of the exposure was only 2 hr. The differences between the spectral reflectance curves for the cherry bond, before and after fading, are listed in Table II.

During the course of the present work, the Vitrolite standards suffered an appreciable drop in reflectance relative to the magnesium oxide scale (which is "stabilized" through the use of a number of aged, carefully stored ceramic standards). The instrument was employed intermittently (considerably less than daily because of the press of other projects) over a period of about 3 mo. During this period, the reflectance of the Vitrolite standards decreased by different amounts at different wavelengths, the maximum decrement



ig. 12. Spectral reflectance of a single pink sheet over lack (1 B); of two sheets over black (2 B), etc., to five heets over black (5 B); and of a sufficient number of sheets to yield R_∞ (GERS)

ccurring at the violet end and in the middle portion f the visible spectrum, where the changes were about -1%. It is often assumed that glass or glasslike aterials are immutable in their reflectance; expeence in our laboratories has shown that standards repared from such materials are stable only after ging and solarization, and then stability is only a elative term—the standards must be checked and revaluated at regular intervals. New standards for he brightness testers are exposed to direct sunlight for n interval of time before the work of standardization begun.

nfluence of Aging on Color Temperature and Sphere Constant

Two effects of potential importance could not be xplored because of time limitations (calendar-wise).

able II. Effect of a 2-Hr. Use of a Paper Reference Standard

Magnesium Oxide Scale

Wavelength, mmu	ΔR, %	*Wavelength, mmu*	ΔR, %	*Wavelength, mmu*	ΔR, %
400	0.0	520	+0.1	600	0.0
420	0.0	540	+0.6	610	−0.5
430	−0.6	550	+1.0	620	−0.9
440	−1.2	560	+1.3	630	−0.7
450	−1.3	570	+1.4	640	−0.6
460	−1.0	580	+1.2	660	−0.2
480	−0.6	590	+0.6	680	−0.3
500	−0.1			700	−0.5

One was the influence of the age of the lamp on the color temperature (or, more to the point, the relative spectral energy distribution), and the other was the influence of age on the sphere constant. The operator is instructed to adjust the lamp voltage from time to time and this is, of course, a desirable instruction. The lamp life is said to be about 6 mo.; however, whatever the lamp life, it is general experience that, in the last stages of the existence of the tungsten filament, the color temperature decreases quite seriously, even when the lamp voltage is held constant. In addition, the envelope of a small-tube incandescent lamp becomes dark at some time prior to the point of burn-out and, although we do not know the specific influence of the dark film on the relative spectral energy distribution, it is our common experience that this film has sufficient spectral effect to impair the accuracy of brightness measurement (the arrangement of the lamp in a G. E. reflection meter is such that the first darkening appears on the side of the lamp facing the condensing lenses—when the lamp is rotated 180°, clear glass is brought into play, and the difficulty is removed). The presumption is that a user of the Color-Eye would not change the lamp until it burns out; good practice in this field of work is to discard a lamp at a much earlier time than the burn-out point—often when the envelope becomes noticeably darkened.

The inside surface of the integrating sphere is painted with ten coatings of a special paint which is said by the manufacturers to be neutral and stable, and to have a reflectance of 94% relative to magnesium oxide (i.e., about 0.91 to 0.92 absolute reflectance). It is impor-

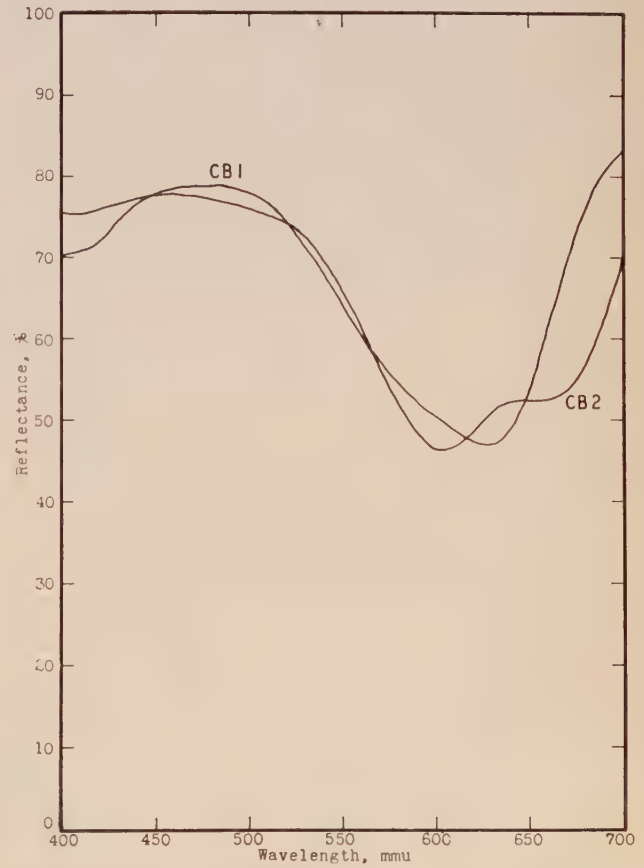


Fig. 13. Spectral reflectance of two coated blue papers (GERS)

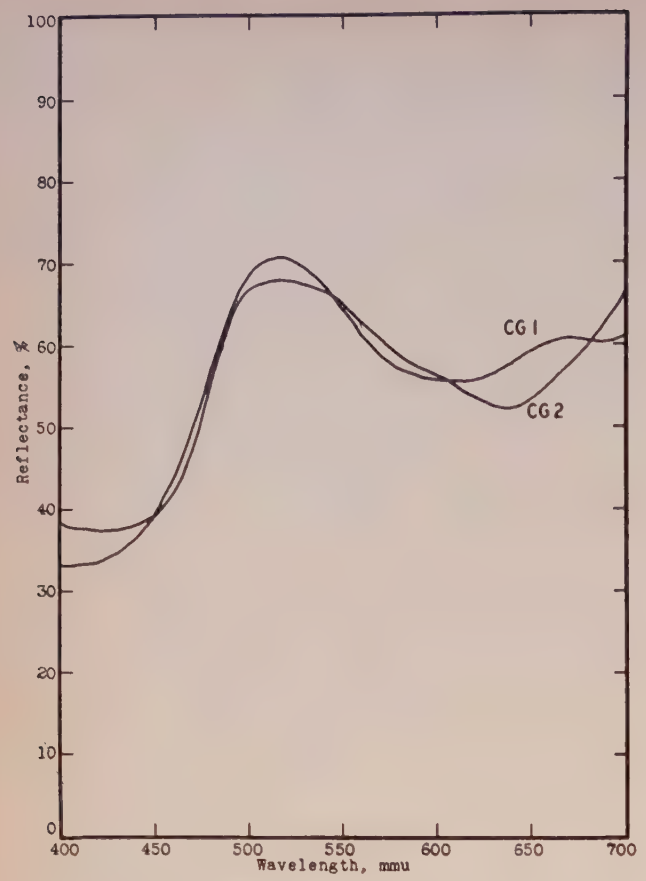


Fig. 14. Spectral reflectance of two coated green papers (GERS)

tant to the long-range *precision* of a filter colorimeter employing an integrating sphere—with regard to reproducibility of color measurements—that the *absolute* reflectance of the sphere wall remain constant, or change in such manner that $(1-R)$ would change by a nearly uniform percentage at all wavelengths (where R is the absolute reflectance of the sphere wall). This follows from the important consideration that the light intensity on the specimen and standard are almost inversely proportional to $(1-R)$. Since R is not much less than 1.0, the quantity $(1-R)$ is small, and only moderate changes in R will produce significant *percentage* changes in $(1-R)$ and, hence, also in the intensity of illumination. Thus, if after long use of a filter colorimeter involving an integrating sphere the sphere wall becomes slightly yellowish, the illumination of the specimen and standard may become undesirably yellowish. Although this could not be checked in the present study, it is mentioned in order that users of the Color-Eye may be aleviated to a possible source of difficulty in work requiring long-range precision.

It is agreed that small changes in color temperature of the light or small percentage changes in $(1-R)$ over the spectrum will not result in appreciable error in the measurement of color differences. Bothersome errors will probably result in those uses of the instrument in which individual values of reflectance are important and, if the yellowing of light in the sphere becomes appreciable, error will also result in the determination of color differences for certain types of color differentials.

PERFORMANCE OF THE COLOR-EYE IN COLOR MATCHING

As in past studies of filter colorimeters, it was considered desirable to compare the performance of the instrument under study with ICI data obtained through the careful use of the G.E. recording spectrophotometer (GERS) and accurate, continuous integration of the spectral products, $RE_C\bar{x}$, $RE_C\bar{y}$, $RE_C\bar{z}$, where the quantities have their usual meanings (*5*), and of similar quantities involving transmittance instead of reflectance. In the present study, the trichromatic coefficients are also compared and the nature of the various chromaticity differences is shown graphically by plotting on the ICI chromaticity diagram (a chart presenting y versus x). In this study we are primarily concerned with a comparison of the chromaticity differences (or "vectors") appearing on the diagram for, if a filter colorimeter is to be of aid in color matching and in other aspects of color control, it should reliably reveal the direction and magnitude of the vector on the chromaticity diagram which runs from one point to the other (and, in addition, the component ΔY should be correct).

In the interests of accuracy of comparison, data have been obtained for families of dye solutions, for several glass color filters, and for a series of papers so chosen that, within each family of papers, the surfaces and "finishes" are nearly identical.

Colorimetry of Dye Solutions and Glass Color Filters

Solutions of Auramine were prepared in four con-

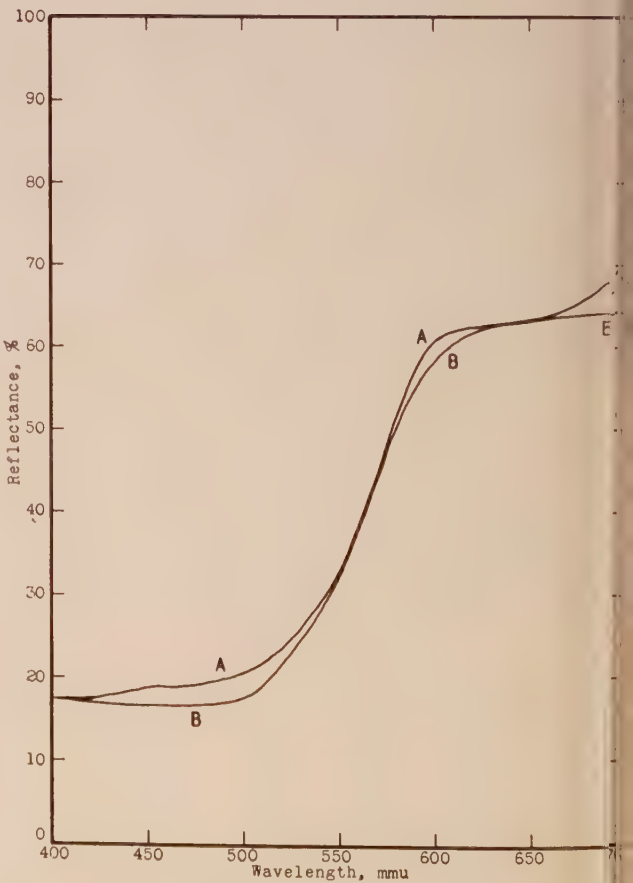


Fig. 15. Spectral reflectance of two of a series of [illegible] coated orange-brown papers (see also Fig. 16) (GERS)

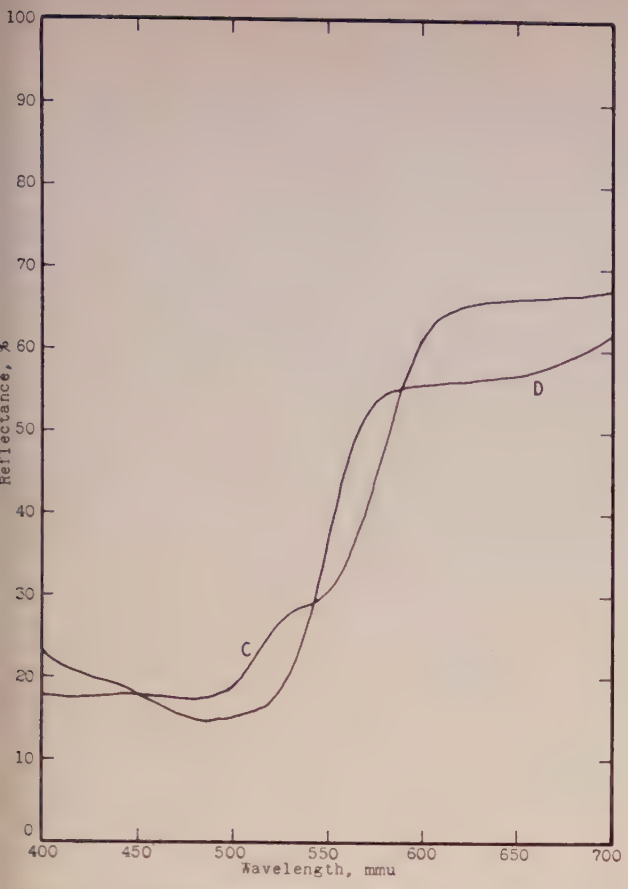


Fig. 16. Spectral reflectance of two of a series of four coated orange-brown papers (see also Fig. 15) (GERS)

modified slightly to 1.1812. (With regard to nomenclature, it would be less confusing to the reader if the manufacturers of the Color-Eye would employ symbols like T_X, T_Y, and T_Z, and R_X, R_Y, and R_Z, instead of X_{CE}, Y_{CE}, and Z_{CE}, which latter are not, and are not claimed to be, tristimulus values.)

The last three columns of Table III present the errors in the Color-Eye tristimulus values. These were calculated to provide information on the question of whether the Color-Eye may be used as a rapid analog computer for the determination of tristimulus values. Although the manufacturers do not stress this application of the Color-Eye, means are presented in the instruction manual for the calculation of ICI tristimulus values. It is there stated (page 19 of the manual) that "The characteristics of the photosensitive surface in the multiplier and the filters are chosen to approximate, as closely as possible, the standard ICI curves. As noted earlier, filter colorimetry can never be perfect due to limitations in filter and photosensitive materials. However, a good approximation can be obtained."

In judging the errors it should be kept in mind that, as a rough rule, a least perceptible difference in color (to a well-trained eye under optimum viewing conditions) is often of the order of 0.5 tristimulus unit (particularly when the variation is primarily in only one tristimulus value, or when a variation in one tristimulus value is accompanied by a change of opposite sign in another tristimulus value). Thus, if filter colorimetry is to present tristimulus values in good approximation to the requirements—i.e., if one wishes to regard such an instrument as an analog computer for the purposes

entrations, of Chrysoidine in four concentrations, of Victoria green in four concentrations, and of Fast red BL and copper sulphate in three concentrations each. In each case, a solution was contained in an optical cell which was inserted alternately in the GERS and the Color-Eye (using the transmission slide). In each instrument, a nearly identical cell containing distilled water was placed alternately in the standard beams of the GERS and the Color-Eye. Transmittances of a blue glass filter, of a yellow glass filter, of a Corning no. 038 filter, and of a Corning no. 3962 filter were similarly obtained in the two instruments; in each case, a plain (uncolored) glass filter was used in the standard beam.

The spectral transmittance curves for the solutions and glass filters are given in Figs. 2 to 8. The tristimulus values obtained by integrations involving the curves given in these figures are included in Table III, which presents also the primary data obtained with the Color-Eye, and the ICI tristimulus values calculated from the Color-Eye readings. For the latter calculations, the manufacturers recommended the following equations, in which X_{CE}, Y_{CE}, and Z_{CE} are the transmittance readings of the Color-Eye with the amber, green, and blue filters, respectively.

$$\begin{aligned} X_{ICI} &= 0.783X_{CE} + 0.197Z_{CE} \\ Y_{ICI} &= Y_{CE} \\ Z_{ICI} &= 1.178Z_{CE} \end{aligned} \qquad (1)$$

These equations approximate those usually given for three-filter colorimeters. In arriving at the data given in Table III, the factor in the last relationship was

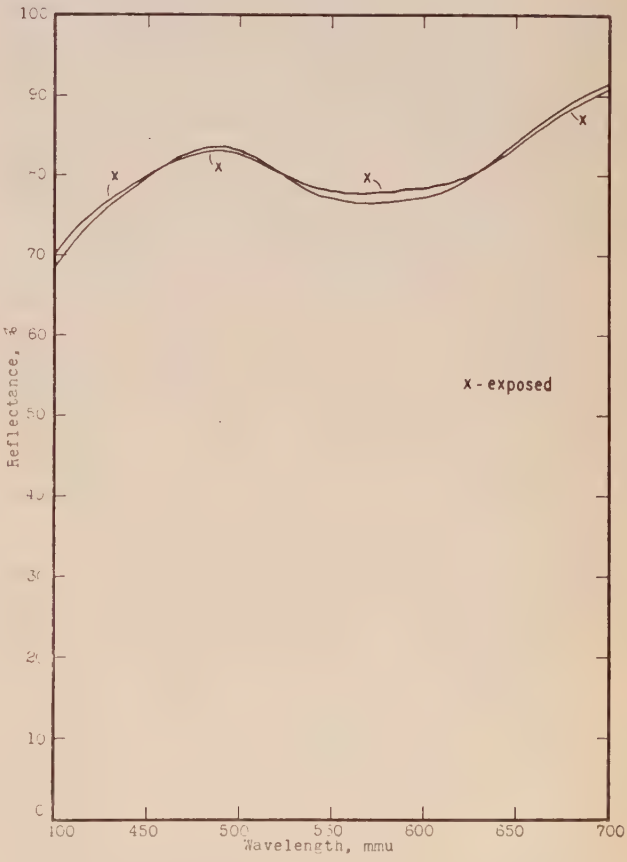


Fig. 17. Spectral reflectance of white bond paper before and after fading on the Color-Eye (GERS)

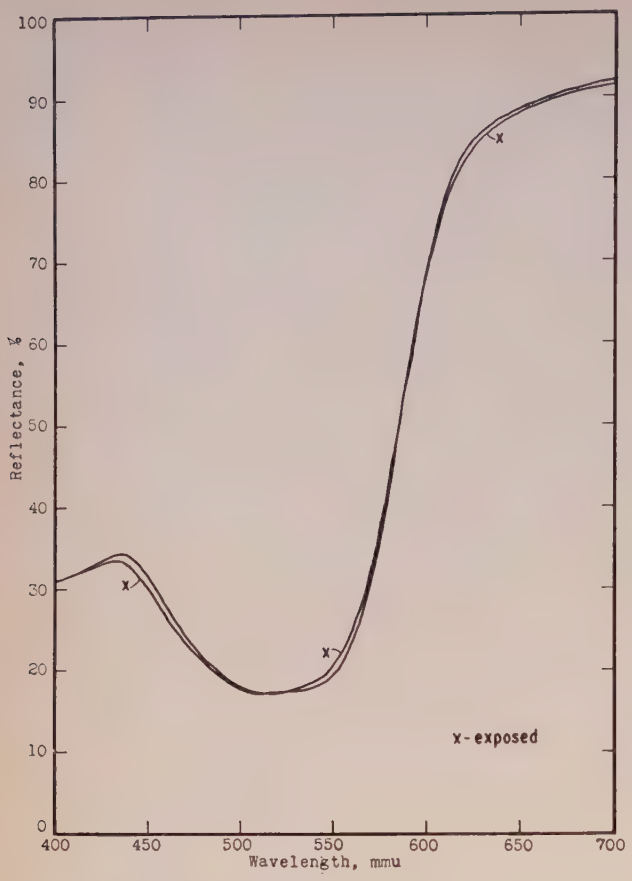


Fig. 18. Spectral reflectance of cherry bond paper before and after fading on the Color-Eye (GERS)

of ICI calculations—the error should be of the order of 0.5 unit, and preferably smaller. Inspection of the errors for the range of colors studied (varying from mild to strong colors) shows that the error in X ranged from -6.1 to $+9.2$, with an average magnitude of 3.1 units; for Y the range was -1.6 to $+4.4$, with an average magnitude of 1.2 units; for Z the range was -5.1 to $+8.6$, with an average magnitude of 3.5 units.

The trichromatic coefficients corresponding to the two sets of ICI tristimulus values in Table III are given in Table IV.

The data presented in Table IV confirm the earlier remarks, based on the tristimulus values, that the Color-Eye is not sufficiently accurate to be used as a means for obtaining ICI data. The length of an error segment on the ICI chromaticity diagram (plot of y versus x) is, of course, the square root of the sum of of the squares of the last two figures given in any row. The largest of the segments corresponding to the data in Table IV is 0.038. This corresponds to a very large color difference. MacAdam (*6*, *7*) and others have obtained data which enable one to estimate, for various parts of the chromaticity diagram and for any direction on the diagram, the standard error of color matching. It seems pointless, in the present instance, to calculate the number of standard errors corresponding to the various errors listed in the table (from which the number of just perceptible chromaticity steps could be estimated), because many of the segments may be seen by inspection to correspond to several just perceptible differences, and about a third of the segments correspond to very appreciable color differences. (As a very rough and ready rule, a segment on the diagram, in terms of the units of Table IV, of length 0.001 corresponds to a perceptible color step.) In studying the data of Table IV, account should be taken of the fact that, for the solutions, as the dilution increased, the chromaticity approached the "white point" for ICI Illuminant C, at which the instrumental error must of necessity (barring fluorescent effects) be essentially zero. The implications of some of the data with regard to the use of the instrument for the measurement of color differences are discussed in later paragraphs of this section.

It is of interest to discuss the nature of some of the instrumental divergences displayed by the transmittances X_{CE}, Y_{CE}, and Z_{CE} for the dye solutions and the glass color filters (Table III). Broadly speaking, the errors in X_{ICI}, Y_{ICI}, and Z_{ICI} are the result of the use of three filters instead of four (*4*), and in the rather bad mismatch of the spectral response of the Color-Eye, for each filter, to the theoretical requirements (see Instruction Manual I.M. 5002, Fig. II-4). Part of the difficulty lies in the photometric error (see section on General Observations). Some of the contributing reasons for the errors in the last three columns of Table III are given with the hope that they may aid in improvement of the design of the Color-Eye. These will be discussed for each family of dye solutions and for each glass color filter.

Auramine (Fig. 2). X_{CE}, as expected, is close to

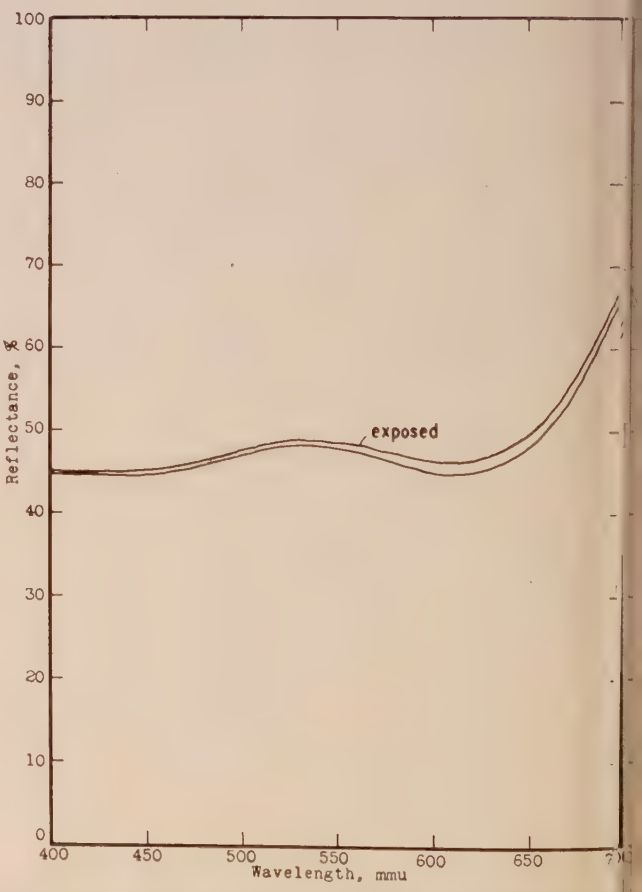


Fig. 19. Spectral reflectance of gray bond paper before and after fading on the Color-Eye (GERS)

00%, so that ΔX is attributable to the error in Z_{CE} ut is about twice the expected error ($0.2 \times 8.2 = 1.6$); his is because the Z-function does not fit (in relative nits) the blue loop of the X-function. Y_{CE} is too arge because of relatively too great emphasis in the pectral response on longer wavelengths (hereafter his will be referred to as "too great centroid wave-ength"). Z_{CE} is much too high, partly because of oo great centroid wavelength and partly because of a robable ultraviolet response (see discussion under hrysoidine); it should be noted that an error of +2.6 or Z_{ICI} at 104.3 is relatively quite substantial, for he photometrical error here would be negligible.

Chrysoidine (Fig. 3). X_{CE} is close to 100% and, ence, the error is small. Y_{CE} is too high because of oo great centroid wavelength. Z_{CE} is much too high; n comparing the spectral transmittance curves of igs. 2 and 3, one sees the probability that the larger rror in the Color-Eye data for Z_{ICI} of Auramine is ttributable to a response of the instrument to near-ltraviolet light.

Victoria green (Fig. 4). X_{CE} is much too great be-ause of too long effective wavelength, particularly as a esult of the Color-Eye's abnormally high response to ed light. The data for Y_{CE} show that the centroid vavelength is too great; the photometric error com-ensates to some extent, diminishing the negative error t the greatest concentration (dilution no. 1). The ositive error at the greatest dilution is not understood the photometric error should here be negligible), un-ess the spectral response of the Color-Eye for the Y-unction includes near-infrared sensitivity of just the mall amount to account for the error. Z_{CE} is much oo high, because of too great centroid wavelength and ossibly because of response to near-ultraviolet light.

Fast red 8BL (Fig. 5). X_{CE} and Y_{CE} are too high ecause of too large centroid wavelengths. Z_{CE} is oo small because of too great centroid wavelength the slope of the spectral transmittance curve is nega-ive); the error would be more strongly negative were t not for the compensating effect of the photometric rror.

Copper sulphate (Fig. 6). X_{CE} is much too small because the centroid wavelength is too great (negative slope of transmittance curves). Y_{CE} is too low because of too great centroid wavelength. Z_{CE} is too near 100% to involve appreciable error.

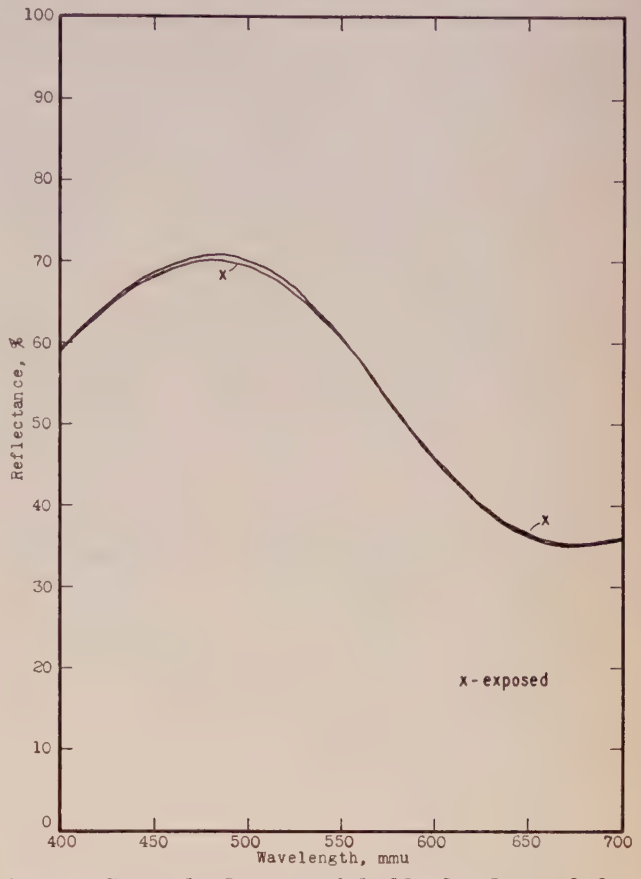


Fig. 20. Spectral reflectance of the blue bond paper before and after fading on the Color-Eye (GERS)

Blue filter (Fig. 7). X_{CE} is too high because of the abnormally high red and near-infrared response of

Table III. Tristimulus Data Obtained with the GERS and Color-Eye

Dye Solutions and Glass Filters

Specimen	*Dilution No.*	*GERS* X_{ICI}	*GERS* Y_{ICI}	*GERS* Z_{ICI}	*Color-Eye* X_{CE}	*Color-Eye* Y_{CE}	*Color-Eye* Z_{CE}	*Color-Eye* X_{ICI}	*Color-Eye* Y_{ICI}	*Color-Eye* Z_{ICI}	*Color-Eye* ΔX	*Color-Eye* ΔY	*Color-Eye* ΔZ
uramine	1	86.0	97.7	58.3	99.8	98.9	56.3	89.2	98.9	66.5	+3.2	+1.2	+8.2
	2	90.9	98.5	85.3	99.8	99.4	76.6	93.2	99.4	90.5	+2.3	+0.9	+5.2
	3	93.3	98.8	96.2	99.9	99.6	84.0	94.9	99.6	100.3	+1.6	+0.8	+4.1
	4	94.8	99.0	104.3	99.9	99.6	90.5	96.0	99.6	106.9	+1.2	+0.6	+2.6
hrysoidine	1	83.8	92.4	40.1	99.2	94.1	38.6	85.3	94.1	45.6	+1.5	+1.7	+5.5
	2	88.9	95.9	67.4	99.3	96.4	60.4	89.7	96.4	71.3	+0.8	+0.5	+3.9
	3	92.8	97.8	88.2	99.5	97.7	76.5	93.0	97.7	90.4	+0.2	+0.1	+2.2
	4	95.1	99.0	101.7	99.6	98.5	86.7	95.1	98.5	102.4	0.0	−0.5	+0.7
ictoria green	1	36.9	49.6	94.4	37.5	49.5	84.9	46.1	49.5	100.3	+9.2	−0.1	+5.9
	2	61.9	72.1	106.5	64.3	73.4	93.0	68.7	73.4	109.9	+6.8	+1.3	+3.4
	3	71.6	79.8	110.5	74.2	81.5	97.0	77.2	81.5	114.6	+5.6	+1.7	+4.1
	4	82.6	88.6	114.5	85.9	90.3	99.4	86.8	90.3	117.4	+4.2	+1.7	+2.9
ast red 8BL	1	71.0	52.7	58.7	83.3	56.2	49.6	75.0	56.2	58.6	+4.0	+3.5	−0.1
	2	82.7	71.6	83.9	90.6	74.0	70.7	84.9	74.0	83.5	+2.2	+2.4	−0.4
	3	89.9	84.7	99.5	95 0	85 9	84.2	91 0	85.9	99.5	+1.1	+1.2	0.0
opper sulphate	1	85.0	91.8	118.0	75.8	91.0	99.4	78.9	91.0	117.4	−6.1	−0.8	−0.6
	2	90.9	95.7	118.0	86 0	95.5	99.9	86.9	95.5	118.0	−4.0	−0.2	0.0
	3	94.0	97.7	118.0	91.1	96.1	99.4	90.9	96.1	117.4	−3.1	−1.6	−0.6
lue filter	..	10.8	3.0	55.2	4.7	2.9	42.4	12.0	2.9	50.1	+1.2	−0.1	−5.1
ellow filter	..	83.0	90.5	59.0	98.7	94.9	57.0	88.5	94.9	67.3	+5.5	+4.4	+8.3
orning No. 038	..	85.8	96.7	64.9	98.0	97.5	62.2	89.0	97.5	73.5	+3.2	+0.8	+8.6
orning No. 3962	..	43.8	50.5	55.8	41.4	51.5	50.3	42.3	51.5	59.4	−1.5	+1.0	+3.6

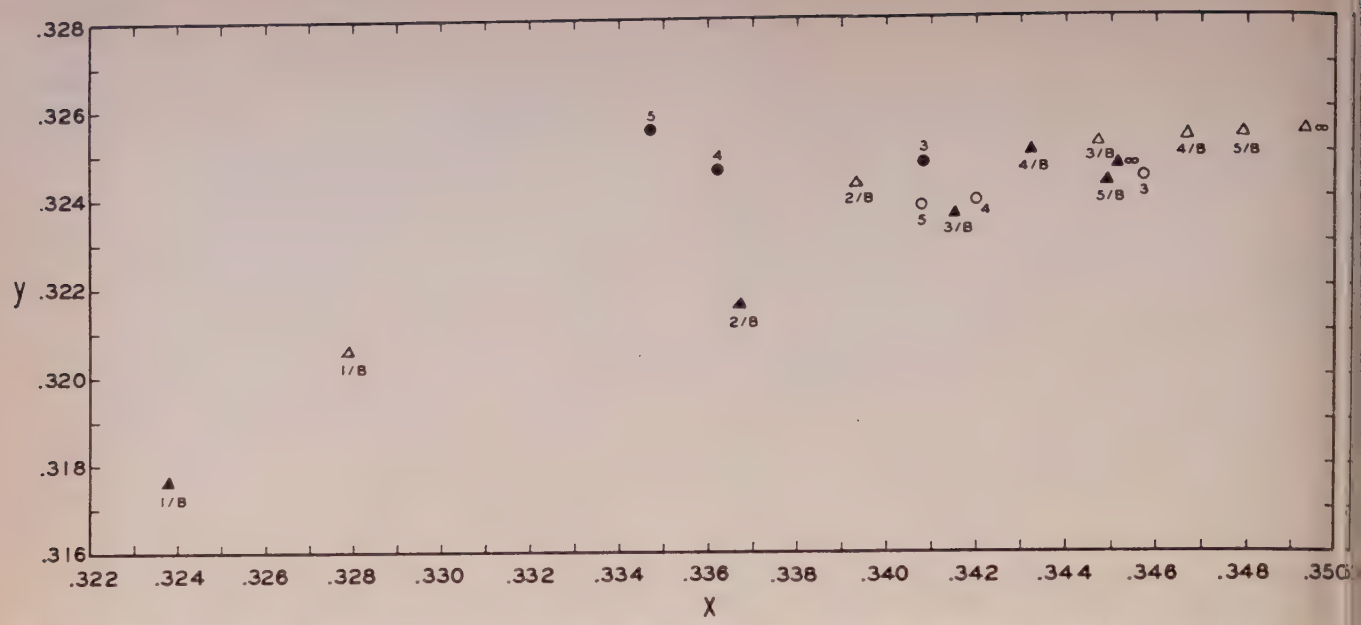


Fig. 21. A portion of the ICI chromaticity diagram giving the x, y plot of the members of the two pink series, based on observations with both the GERS (solid points) and the Color-Eye (open points)

the Color-Eye when used with the amber filter; on the basis of equations (1), using the correct ICI data, X_{CE} should be 2.1 and, hence, the observed value (4.7) is high by 2.6. Y_{CE} is almost exactly correct. Z_{CE} is seriously low because the centroid wavelength is too great (the transmittance curve has negative slope).

Yellow filter (Fig. 7). X_{CE} is too high, partly because the centroid wavelength is too long. Y_{CE} is appreciably too high, in spite of the nearness of the transmittance to 100%, because the centroid wavelength is too long. Z_{CE} is substantially too great because the centroid wavelength is too long.

Corning No. 038 (Fig. 8). X_{CE} is too high, partly because the centroid wavelength is too great. Y_{CE} is a little too high (the transmittance is near 100%). Z_{CE} is much too high because of the abnormally high response of the instrument over the right half of the Z-function.

Corning No. 3962 (Fig. 8). X_{CE} is too low, in spite of the compensating photometric error, because the centroid wavelength is too long. Y_{CE} is too high, possibly because of the photometric error. Z_{CE} is too high because of the too great centroid wavelength.

The consistency of the relationships between the stated sources of error and the observed errors is convincing. Of course, an exact analysis of the errors should not rest on the concept of the "centroid wavelength" (first moment of a function) but, rather, on the complete functions and their integrals.

Colorimetry of Paper

Instrumental comparisons based on data from spectral transmittance (foregoing subsection) are ideal in the sense that uncertainities arising from differences in geometries of illuminating and viewing are not involved. However, the situation is different in the comparison of spectral reflectance. It is an age-old fact that the color one perceives on viewing a colored body (by reflected light) depends on the orientation of the object with respect both to the light source and the eye. Similarly, instrumental reflectance depends numer-

Table IV. Comparison of Trichromatic Coefficients

Specimen	*Dilution No.*	*GERS* x	*GERS* y	*Color-Eye* x	*Color-Eye* y	*Color-Eye* Δx	*Color-Eye* Δy
Auramine	1	0.3553	0.4038	0.3504	0.3885	−0.0049	−0.0153
	2	0.3311	0.3584	0.3292	0.3511	−0.0019	−0.0073
	3	0.3235	0.3427	0.3219	0.3379	−0.0016	−0.0048
	4	0.3180	0.3322	0.3174	0.3293	−0.0006	−0.0029
Chrysoidine	1	0.3877	0.4271	0.3791	0.4182	−0.0086	−0.0089
	2	0.3524	0.3802	0.3485	0.3745	−0.0039	−0.0057
	3	0.3329	0.3507	0.3308	0.3476	−0.0021	−0.0031
	4	0.3216	0.3346	0.3213	0.3328	−0.0003	−0.0018
Victoria green	1	0.2038	0.2744	0.2353	0.2527	+0.0315	−0.0217
	2	0.2576	0.2997	0.2726	0.2913	+0.0150	−0.0084
	3	0.2734	0.3047	0.2825	0.2982	+0.0091	−0.0065
	4	0.2892	0.3102	0.2947	0.3066	+0.0055	−0.0036
Fast red 8BL	1	0.3890	0.2889	0.3952	0.2961	+0.0062	+0.0072
	2	0.3473	0.3005	0.3502	0.3053	+0.0029	+0.0048
	3	0.3280	0.3090	0.3292	0.3108	+0.0012	+0.0018
Copper sulphate	1	0.2883	0.3116	0.2746	0.3167	−0.0137	+0.0051
	2	0.2985	0.3140	0.2894	0.3177	−0.0091	+0.0037
	3	0.3036	0.3155	0.2986	0.3157	−0.0050	+0.0002
Blue filter	..	0.1567	0.0435	0.1846	0.0446	+0.0279	+0.0011
Yellow filter	..	0.3570	0.3891	0.3530	0.3785	−0.0040	−0.0106
Corning No. 038	..	0.3468	0.3909	0.3423	0.3750	−0.0045	−0.0159
Corning No. 3962	..	0.2917	0.3362	0.2761	0.3362	−0.0156	0.0000

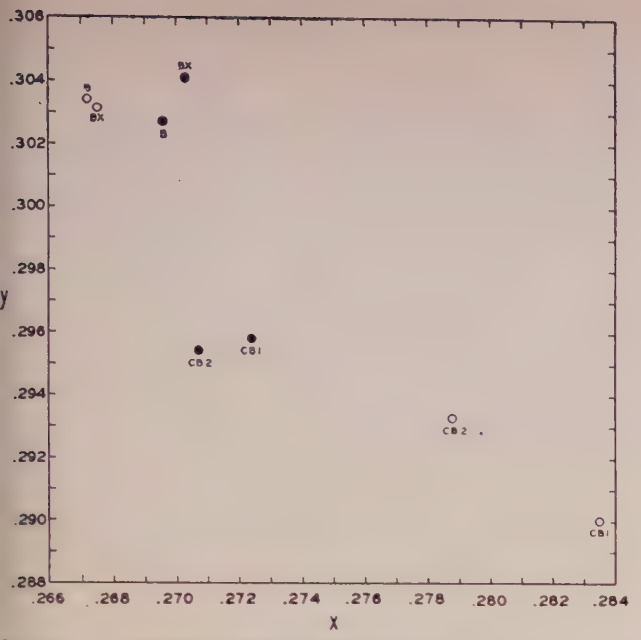


Fig. 22. A portion of the ICI chromaticity diagram giving the x, y plot of the two coated blue papers (CB 1 and CB 2) and the blue bond paper before and after fading (B and BX) based on observations with both the GERS (solid points) and the Color-Eye (open points)

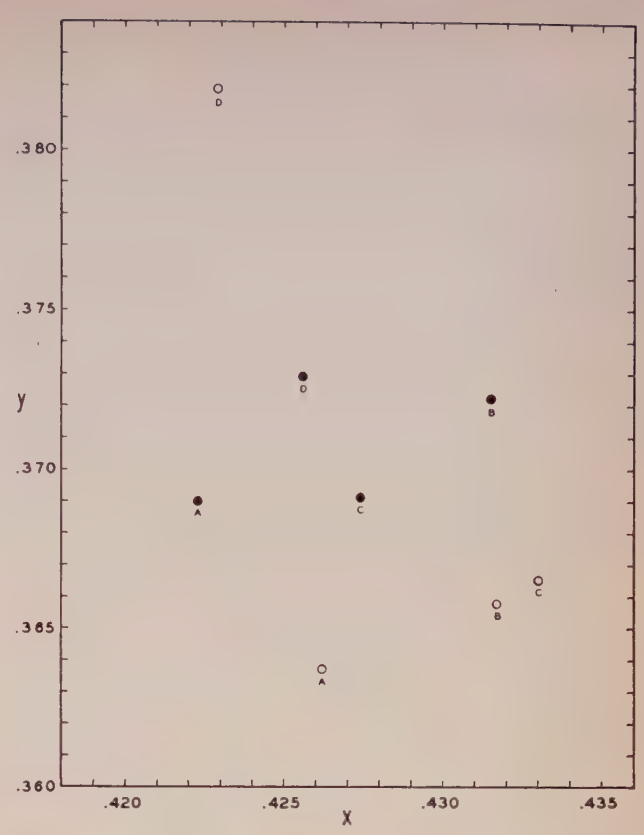


Fig. 23. A portion of the ICI chromaticity diagram giving the x, y plot of the members of the coated orange-brown papers, based on observations with both the GERS (solid points) and the Color-Eye (open points)

ally upon the geometry of illuminating and viewing. The geometry of the Color-Eye is not quite the reciprocal of that of the GERS, because the light accepted for measurement leaves the specimen and the standard at 40° rather than at 0°, and there are other differences. Therefore, a comparison of reflectance data analogous to that made in the foregoing subsection of transmittance data would be subject to uncertainty of interpretation. There would, nevertheless, be interest in a comparison of colorimetric data obtained with the two instruments. The effect of geometry is rarely ever very large (in the intermediate range of reflectances of paper, the rather large change from the ICI 45° and 0° mode of illuminating and viewing to the use of an integrating sphere is at the most about 5%). For papers of the same grade and of similar "finish" and surface texture, the differences in geometries should not cause appreciable error in the comparison of color differences evaluated by means of the GERS and the Color-Eye.

Data obtained with the Color-Eye for commercial papers showed that there is a directional effect in the sense that reflectances observed when the machine direction of the paper is vertical are generally greater than the values read when the machine direction is horizontal. Accordingly, data were obtained for both directions and averages are given in this report; the differences between the reflectances observed for the two orientations varied from 0.2 to 0.6% reflectance.

The Color-Eye results for the ICI tristimulus values for a miscellaneous collection of white and colored bond papers of similar manufacture and "finish" are given in Table V (GERS spectral reflectivity curves in Figs. 9 and 10), and the tristimulus values for nine series of papers representing small to large color differences are given in Table VI (GERS spectral reflectance curves in Figs. 11 to 20), together with the indicated errors in the trichromatic coefficients. The significance of the errors in the trichromatic coefficients is discussed in the next subsection. In the interests of objectivity, reflectance data were obtained with the Color-Eye using the local magnesium oxide scale of reflectance and the values supplied by the manufacturers for the Vitrolite standards; as slightly better agreement was obtained through the use of the latter, only the data obtained with the Vitrolite standards are given in Tables V and VI.

The ICI tristimulus values calculated from the Color-Eye reflectances were obtained through the use of the equations suggested by the manufacturers (these include the manufacturers' values for the Vitrolite standards):

Table V. Tristimulus Values and Trichromatic Coefficients Obtained with the GERS and Color-Eye

Miscellaneous Papers

Specimen of bond paper	*GERS*					*Color-Eye*						
	XICI	YICI	ZICI	x	y	XICI	YICI	ZICI	x	y	Δx	Δy
Gray	44.8	46.7	52.8	0.3105	0.3236	46.8	46.7	52.2	0.3212	0.3205	+0.0107	−0.0031
White	76.4	77.9	93.2	0.3088	0.3147	76.0	75.9	90.4	0.3137	0.3132	+0.0049	−0.0015
Primrose	66.1	72.8	56.1	0.3389	0.3734	64.3	71.1	59.3	0.3303	0.3652	−0.0086	−0.0082
Russet	45.0	43.0	26.7	0.3924	0.3749	45.6	44.4	29.3	0.3822	0.3722	−0.0102	−0.0027
Cherry	49.9	34.9	34.1	0.4196	0.2937	52.3	34.3	30.1	0.4482	0.2939	+0.0286	+0.0002
Green	41.8	50.8	58.6	0.2766	0.3357	39.4	48.9	58.1	0.2691	0.3340	−0.0075	−0.0017
Blue	51.1	57.4	80.5	0.2704	0.3036	48.7	55.3	78.3	0.2671	0.3033	−0.0033	−0.0003

$$X_{ICI} = 0.6850X_{CE} + 0.1678Z_{CE}$$
$$Y_{ICI} = 0.8764Y_{CE} \qquad (2)$$
$$Z_{ICI} = 1.0229Z_{CE}$$

A detailed discussion of the data presented in Table V would roughly parallel that for the data given in Tables III and IV for the dye solutions and glass color filters. The segments of the ICI chromaticity diagram representing the chromaticity errors of the Color-Eye are quite large, although allowance must be made for the fact that some chromaticity difference is not the result of error but a consequence of the differences between the geometries of illuminating and viewing of the GERS and the Color-Eye. As a rule, however, surface reflection effects are fairly achromatic, and large differences in instrumentally determined chromaticities are not to be expected when the geometries are as similar as those of the two instruments here involved. In any event, the data presented in Table V are more for the purposes of providing general information than for analysis.

Measurement of Color Differences

Some of the data presented in Tables III and IV may be used to test the Color-Eye for accuracy in measuring color differences for which only one, and the same, colorant is involved. This condition is not, however, a realistic simulation of the color differences encountered in color matching and color control work in the paper industry. The color differences are usually small and, even where the furnish and dyestuffs are the same, nearly all the near-matches are metameric in one degree or another. Accordingly, colorimetric data have been obtained for several series of papers: a series of well-aged dyed handsheets (pink) having almost identical surface finish (Fig. 11); a series comprised of (1) a single pink handsheet backed by black, (2) two pink sheets backed by black, (3) three sheets backed by black, (4) four sheets backed by black, (5) five sheets backed by black, and (6) a sufficient number of sheets to yield R_∞ (Fig. 12); two coated papers (blue) representing a typical attempt to match an unknown (Fig. 13); two coated papers (green) representing a typical attempt to match an unknown (Fig. 14); a series of four coated papers (orange brown) containing different pigments and dyestuffs (Figs. 15 and 16); a commercial white bond before and after a 2.75-hr. exposure over a sphere port of the Color-Eye (Fig. 17); a commercial cherry bond before and after a 2-hr. exposure on the Color-Eye (Fig. 18); a commercial gray bond before and after a 2-hr. exposure on the Color-Eye (Fig. 19); a commercial blue bond before and after a 2-hr. exposure on the Color-Eye (Fig. 20).

The colorimetric data for these papers, observed with the GERS and the Color-Eye, are presented in Table VI.

Although the accuracy of the GERS or of the Color-Eye does not justify the quotation of trichromatic coefficients to 0.0001, the calculations have been carried to that place, because the manufacturers of the Color-Eye have placed emphasis on the use of their instrument for the exact measurement of color difference. Hence, *precision* in the technical sense is of importance in this study. Great care was exercised to perform measurements with the two instruments (literally side-by-side) on the same areas of the same specimens with the measurements alternating between the instruments. It is estimated that the trichromatic coefficients quoted in columns five and six of Table V are reliable for the purposes of color difference measurements (on the ICI basis) generally to within 0.000 (i.e., the zone of uncertainty around each point on the ICI chromaticity diagram has a radius of about 0.0005). The precision of the x, y values determined with the Color-Eye, from the point of view of reproducibility of result, is probably better than this; a major question of this section is whether the precision of the Color-Eye is meaningful and reliable.

The results for the pink series are shown in Fig. 2. In this and the remaining figures, the solid points represent the x, y values determined with the GERS and the ICI distribution functions of the Standard Observer for Illuminant C. The open points represent the x,

Table VI. Tristimulus Values and Trichromatic Coefficients Obtained with the GERS and Color-Eye
Several Series of Papers for Color Difference Study

Series	GERS					Color-Eye						
	X_{ICI}	Y_{ICI}	Z_{ICI}	x	y	X_{ICI}	Y_{ICI}	Z_{ICI}	x	y	Δx	Δy
Pink, 3	60.7	57.9	59.6	0.3408	0.3247	61.6	57.8	58.8	0.3457	0.3244	+0.0049	−0.0003
Pink, 4	59.6	57.6	60.2	0.3362	0.3246	60.5	57.3	59.1	0.3420	0.3239	+0.0058	−0.0007
Pink, 5	59.1	57.5	60.0	0.3347	0.3255	60.4	57.4	59.4	0.3408	0.3238	+0.0061	−0.0017
Pink, 1/black	47.4	46.5	52.5	0.3238	0.3176	49.4	48.3	53.0	0.3279	0.3206	+0.0041	+0.0030
Pink, 2/black	57.4	54.9	58.3	0.3367	0.3216	58.0	55.5	57.5	0.3393	0.3243	+0.0026	+0.0027
Pink, 3/black	60.1	57.0	58.9	0.3415	0.3236	60.7	57.2	58.1	0.3447	0.3252	+0.0032	+0.0016
Pink, 4/black	60.9	57.7	58.9	0.3432	0.3250	61.5	57.7	58.2	0.3467	0.3253	+0.0035	+0.0003
Pink, 5/black	61.4	57.7	58.9	0.3449	0.3243	61.9	57.9	58.2	0.3479	0.3254	+0.0030	+0.0011
Pink, R_∞	61.6	57.9	58.9	0.3451	0.3247	62.4	58.1	58.2	0.3493	0.3250	+0.0042	+0.0003
Coated, blue	57.6	62.6	91.3	0.2724	0.2958	59.2	60.6	89.1	0.2835	0.2900	+0.0111	−0.0058
Coated, blue	56.8	62.0	91.1	0.2707	0.2954	57.9	60.9	88.8	0.2788	0.2933	+0.0081	−0.0021
Coated, green	53.7	61.7	52.0	0.3208	0.3683	56.3	62.5	56.0	0.3222	0.3576	+0.0014	+0.0107
Coated, green	53.3	61.0	51.7	0.3210	0.3674	55.7	61.8	55.3	0.3221	0.3576	+0.0011	−0.0098
Orange-brown A	45.7	40.0	22.6	0.4223	0.3690	47.3	40.4	23.3	0.4262	0.3637	+0.0039	−0.0053
Orange-brown B	44.5	38.4	20.3	0.4315	0.3722	46.0	39.0	21.6	0.4317	0.3658	+0.0002	−0.0064
Orange-brown C	45.0	38.9	21.4	0.4274	0.3691	48.0	40.7	22.3	0.4330	0.3665	+0.0056	−0.0026
Orange-brown D	44.2	38.8	20.9	0.4256	0.3729	46.4	41.9	21.4	0.4229	0.3819	−0.0027	+0.0090
White, before exp.	76.4	77.9	93.2	0.3088	0.3147	76.0	75.9	90.4	0.3137	0.3132	+0.0049	−0.0015
White, after exp.	77.1	78.5	93.4	0.3096	0.3152	76.5	76.6	90.7	0.3138	0.3141	+0.0042	−0.0011
Cherry, before exp.	49.9	34.9	34.1	0.4196	0.2937	52.3	34.3	30.1	0.4482	0.2939	+0.0286	+0.0002
Cherry, after exp.	49.8	35.3	33.1	0.4216	0.2987	52.2	34.7	29.9	0.4469	0.2971	+0.0253	−0.0016
Gray, before exp.	44.8	46.7	52.8	0.3105	0.3236	46.8	46.7	52.2	0.3212	0.3205	+0.0107	−0.0031
Gray, after exp.	45.8	47.6	53.7	0.3115	0.3236	47.6	47.5	52.8	0.3218	0.3211	+0.0103	−0.0025
Blue, before exp.	51.1	57.4	81.0	0.2696	0.3027	48.7	55.3	78.2	0.2672	0.3034	−0.0024	+0.0007
Blue, after exp.	51.1	57.5	80.5	0.2703	0.3041	48.5	55.0	77.8	0.2675	0.3031	−0.0028	−0.0010

values determined with the Color-Eye. There are two pink series: nos. 3, 4, and 5 (Fig. 11), and 1/B,... 5/B, R_∞ (Fig. 12), the former represented by circles, the latter by triangles. Perhaps the first thing to note (ignoring the absolute errors) is that the open circles are below the trend of the open triangles, whereas the solid circles are above the trend of the solid triangles (here, and in the following, we will use such expressions as the "solid 2/B" in the interest of economy of language). This undoubtedly results from the fact that, although the same colorants were used in the two series, one series was obtained by varying the number of sheets over black, whereas the other was obtained by varying the amounts of the colorants; in consequence, the spectral reflectance curves were not closely of the same form. Because of this discrepancy in relative positions of the points, there would be, in some instances, substantial errors in the measurement of color differences by means of the Color-Eye, when members of the series are assigned correct values, and used as standards.

In order that this may be understood, it should be pointed out that, except for huge differences, the use of a colored standard with a filter colorimeter causes the points on the ICI diagram to shift almost, but not quite, in pure translation. The use of such a standard results in new reflectances: $X_{CE}=eX'_{CE}$, $Y'_{CE}=fY_{CE}$ and $Z'_{CE}=gZ_{CE}$, where the factors e, f, and g are not greatly different from unity. Calculations show that the slope of a segment connecting neighboring points changes but little when such a shift is brought about. Therefore, if any pair of points on the diagram are not too far apart, *one may infer what the Color-Eye would indicate for the color difference, if one of the points were made a reference.*

When this technique is applied to the array shown in Fig. 21, it is seen that both nearly reliable and badly unreliable results would be obtained. The points 1/B might be omitted as being too remote from the general cluster. Examples: (1) If 3/B is used as standard, shifting open 3/B to solid 3/B results in fair accuracy of locating the other points (error segments typically of the order of 0.001), if, for the moment, the series designated with circles is ignored. (2) If 2/B is made the standard, the location of other points (considering now all the points) may be very badly in error; e.g., open 4 would be located on the *wrong* side of solid 2/B, and the error of location would be of the order of 0.005, which is substantial, in terms of what trained color men in the paper industry can discern and object to.

Proceeding to the results for the coated blue pair, and using either as a reference, it may be shown (Fig. 22) that the Color-Eye improperly locates the other point with a segment error of 0.005. This near-match for diffuse daylight is strongly metameric (Fig. 13).

The Color-Eye shows the coated green pair (also metameric, Fig. 14) to be an almost exact match, whereas the pair constitute a near-match (segment difference of 0.001).

The results for the orange-brown series (spectral reflectance curves in Figs. 15 and 16) are shown in Fig. 23. The array is typical of the efforts of an experienced man in the industry attempting to meet the color of an unknown sample without the aid of spectrophotometry. The color differences under diffuse daylight are easily noticeable and some of the pairs (of which, of course, there are six) are strongly metameric. Note should be made of the fact that the Color-Eye indicates that B and C nearly match in color; actually (for Illuminant C) the segmental difference is appreciable. Moreover, the Color-Eye indication of the interval from B to C is in the wrong direction. If B or C were used as standard, the segment error would amount to 0.0066. The most favorable case for the instrument would be found in using A as the reference; B would then be improperly located with a segment error of 0.004, and C would be misplaced by 0.003. The result for D is, of course, very badly in error, the segment error being of the order of 0.016.

The results for the white bond paper, before and after fading (Fig. 17), are in agreement for the segmental difference (about 0.0009), but not in the nature of the approach to the white point; part of the latter could be attributed to differences between the magnesium oxide scales at the Institute and at the Instrument Development Laboratories.

The data for the cherry bond, before and after fading (Fig. 18), are in agreement with the fact that the result of fading was easily visible. The ICI segment corresponding to the fading is 0.0054, whereas the Color-Eye segment is 0.0034 and does not lie in the proper direction. Using either the original cherry bond or the faded material as a reference, the segment error for the Color-Eye in this instance is found to be 0.0037, which is felt to be appreciable. In view of the similarity of the spectral reflectance curves and the fact that they simulate either the deviations encountered during the control of color during production or the kind of situation encountered in a fading or aging study, the work was carefully repeated (all work had already been replicated); but, for the second determination, the differences between the spectral reflectance curves were carefully read from the original charts* and, after multiplication by ten, were continuously multiplied by the $E_C\bar{x}$, etc., functions and integrated. In this way, very accurate determinations were made of the changes produced by fading in the ICI tristimulus values.

The segmental difference caused by fading the gray bond paper (Fig. 19), as measured by the Color-Eye, is about the same as that determined with the GERS. It should be noted that, for a neutral color like gray, the absolute values for the trichromatic coefficients determined with the Color-Eye should be subject to less error than indicated by the data given in Table VI; the segment error of about 0.011 is felt to be unduly large. This large error results in this case partly because of the fact that the spectral reflectance curve rises in the red portion of the spectrum (the curves for many dyed papers exhibit this) and because the spectral response of the Color-Eye is too strong toward the red end of the visible spectrum.

The results for the original and the faded blue bond paper (Fig. 20) are included in Fig. 22. It will be seen that the Color-Eye indicated virtually no difference between the original and faded (BX) paper, whereas a difference of possible significance is shown by the ICI data. Of interest is the fact that the neighboring open points for the coated blue papers are disposed

* All the spectral transmittance and reflectance curves given in this report were traced by hand and are subject to error; all data reported were obtained from the original recordings.

relatively to the open B and BX points in a manner different from the disposition of the solid CB1 and CB2 relative to the solid B and BX. A part of this discrepancy may result from the fact that the surfaces of the coated and bond papers are very different, and that the geometries of the two instruments are not the same. This factor is not involved in any of the other examples, for all paper specimens involved in the several series have, within each series, the same finish.

The trichromatic coefficients for the families of dye solutions, given in Table IV, may be employed to test the reliability of the Color-Eye in the measurement of color differences where the colorant is constant, and where the color differences vary from small to large. The method used in the foregoing may be employed for any pair of solutions selected from a family, or alternatively, an equivalent analytical approach may serve as the basis for calculations: The segment error after translation of either point of a pair (corresponding to making one of the pair a reference standard) $= [(\Delta x_q - \Delta x_p)^2 + (\Delta y_q - \Delta y_p)^2]^{1/2}$, in which the quantities involved are those given in the last two columns of Table IV for the pth and qth items. As a matter of numerical convenience, one may work in terms of 0.0001 as a unit. As a numerical example, we may take dilutions nos. 2 and 3 of the Auramine group ($p = 2, q = 3$). The segment error $=0.0001[(-16 + 19)^2 + (-48 + 73)^2]^{1/2}=0.0025$. This particular error is not very large, but is not small enough to be considered satisfactory from the viewpoint of accurate colorimetry. As pointed out earlier in this report, as the concentration of the solutions becomes lower, the chromaticity approaches that of the "white point," at which the error must be zero. Thus, the largest errors will be found for pairs of solutions having the highest concentrations. In the case of the Victoria green family, where the largest error in color difference is indicated, we obtain the following segment errors: 0.0212 for dilutions nos. 1 and 2, using either as a reference; 0.0062 for nos. 2 and 3; 0.0046 for nos. 3 and 4. Of course, this method for estimating errors holds accurately only for small color differences. It was thought to be of interest to examine the matter of the results for the Victoria green family from a more analytical point of view. On assuming that dilution no. 1 were used as a reference standard, it was found by calculation that the Color-Eye transmittances had to be shifted in the following way: X_{CE} had to be modified from 37.5% (see Table III) to 27.1%, so that $e=0.723$; Y_{CE} was modified from 49.5 to 49.6%, so that $f=1.002$; and Z_{CE} was modified from 84.9 to 79.8%, so that $g=0.941$. Now, hypothetically, using dilution no. 1 as a standard, the transmittances for dilution no. 2 were calculated from the observed values, using the factors e, f, and g, and the tristimulus values were obtained; from these the trichromatic coefficients were found: $x'=0.2325$ and $y'=0.3190$. The startling result is that the use of one of the solutions as reference would have led to a greater error in the trichromatic coefficients than that which had been observed using water as a reference. This is probably because of the fairly large gap between the data for dilutions nos. 1 and 2; the large percentage correction necessitated for dilution no. 1 overshadowed the error present in the determination on dilution no. 2. Reversing the situation, and using dilution no. 2 as the reference, it was found by calculation that the trichromatic coefficients which would have been observed for dilution no. 1 were: $x'=0.2232$ and $y'=0.2590$. These are closer to the correct coefficients than those directly observed (see Table IV). The corresponding segment error would be 0.025, which is somewhat larger than the 0.0212 estimated by the method of shifting the points on the ICI diagram in pure translation.

These data, together with others, show that X_{CE} may at times be in very substantial percentage error. Actually, of course, the errors discussed in this section are compounded of spectral errors in all three functions and the photometric error.

GENERAL COMMENT

The manufacturers of filter colorimeters, quite generally, have claimed, over the years, that the spectral response functions for their instruments need to approximate the theoretical requirements only roughly, provided that any color difference under measurement is small. Some have cautioned that the spectral reflectance (or transmittance) curves of the specimen and standard should be very nearly the same, for accurate results. But, unless the color man in a paper mill is equipped with a recording or abridged spectrophotometer, how is he to know that the spectral reflectance of the paper under production differs from standard at all wavelengths by a constant factor (incidentally, a rare and difficulty attainable situation)? How is he to know where to draw the line between a weakly metameric and a strongly metameric match?

There is a need in the paper industry for filter colorimeters which will permit the rapid and reliable checking of color. It is hoped that the manufacturers of such instruments will attack more intensively the analysis of the spectral aspects of these interesting devices and come forth with really reliable colorimeters.

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This report is one of a series issued as a part of the Instrumentation Program of the American Paper and Pulp Association. The data of the present study were reported to the Association as Report 17, Part X, dated Jan. 28, 1952.

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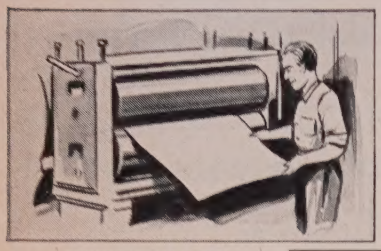

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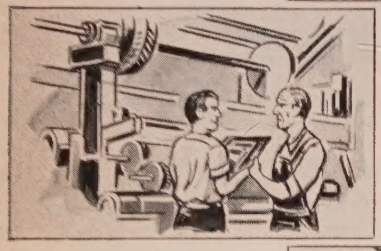

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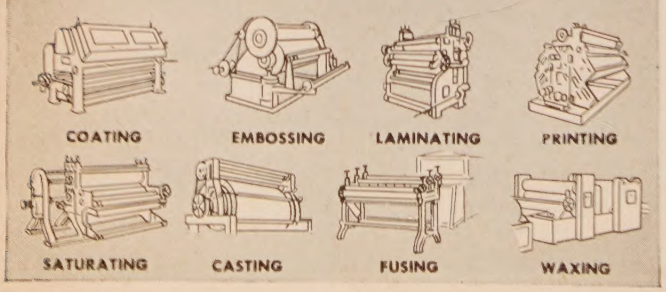

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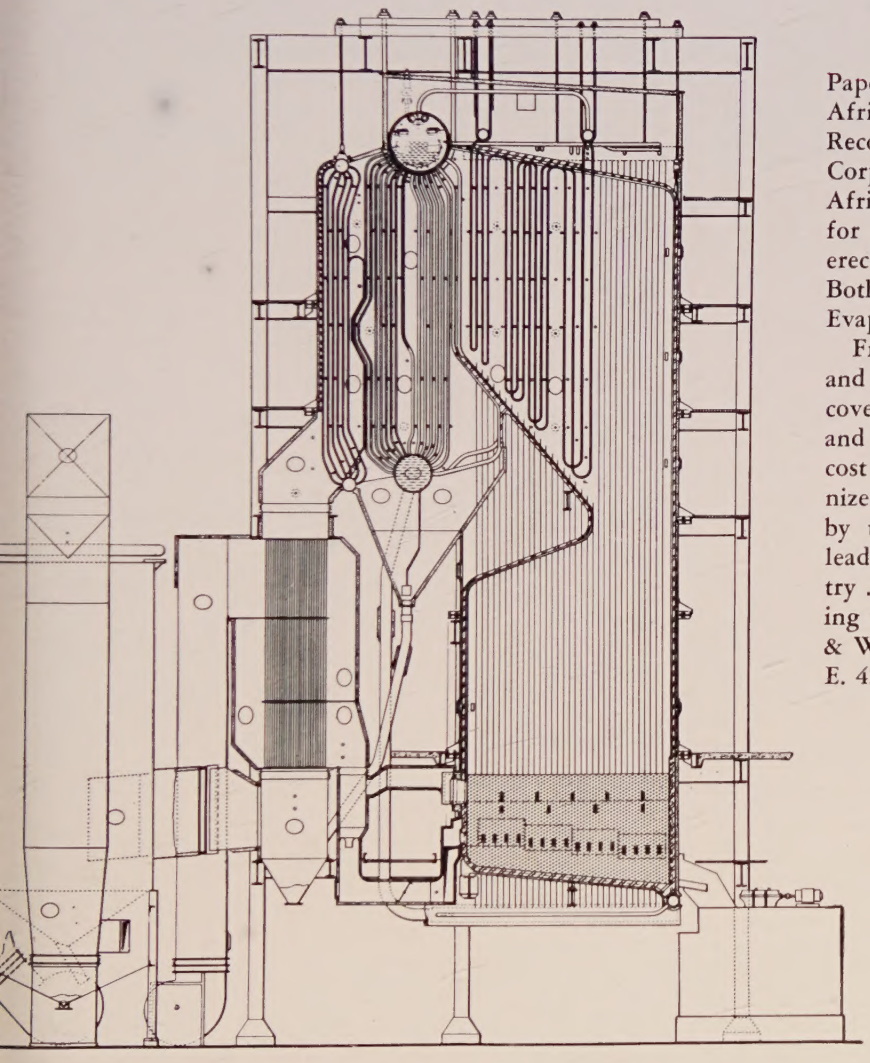

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